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Visual gamma:
eriss gamma analysis technical manual

A Esparon & J Pfitzner

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Executive summary

The Environmental Radioactivity Group (EnRad) of the Supervising Scientist Division (SSD) employ High Purity Germanium (HPGe) gamma detectors to analyse samples for a range of naturally occurring radionuclides and several nuclear bomb fallout radionuclides. The samples are sourced from internal projects and external clients and includes the following:

- Sediments and soils
- Plant and animal material
- Filters from water and dust collection.

Since 1985 computer programs developed in-house have been used for the analysis of the spectrum obtained from the ***eriss*** HPGe detectors. The original programs were written in Fortran. During that time there has been continual improvement of these programs and this latest version, *Visual Gamma*, is the culmination of input from several EnRad staff over those years.

Visual Gamma is written by Andrew Esparon of ***eriss*** in *VisualBasic.net* and incorporates an SQL database and the Ortec Connections software. The Ortec Connections software facilitates communication between the instrumentation and the *Visual Gamma* program. An advantage of the in-house programming is that the source code is available and documented, providing the opportunity for future improvements to the program.

The significant new features of *Visual Gamma* are that it provides a graphical interface, which allows interactive fine tuning of the analysis process by the operator, and a database system for recording sample metadata that is linked to analytical results, therefore allowing efficient data retrieval and manipulation. This document is the technical manual for the *Visual Gamma* computer program.

Visual gamma: *eriss* gamma analysis technical manual

A Esparon & J Pfitzner

1 Scope of *Visual Gamma* and this manual

1.1 Scope

The *Visual Gamma* computer program has been developed for the gamma spectrometry analysis of environmental samples.

Visual Gamma was developed for the analysis of three main categories of gamma spectra: samples, calibration standards and the Quality Assurance suite of stability standards and instrument backgrounds.

Limitations

Visual Gamma is **not** intended for the analysis of samples containing man-made radionuclides other than the following: bomb fallout radionuclides ^{137}Cs, ^{241}Am and ^{60}Co and the manganese precipitation tracer ^{54}Mn.

Note: special calibration may be required for ^{241}Am, ^{60}Co and ^{54}Mn.

It is **not** recommended that *Visual Gamma* be used to analyse gamma spectra saved prior to 2008 using Maestro format (.SPC or .CHN) due to the difficulty of ensuring that the correct calibration data are used for calculations. If anaylsis of historical gamma spectra is required then contact the Program leader of Environmental Radioactivity to ensure correct calibration and background values are used for analysis.

From the one user interface *Visual Gamma* can be used to register samples in the database, to start and stop counting on the HPGe gamma detectors and to save and analyse spectra.

1.2 How to use this manual

This manual contains descriptive information about gamma emission radioactivity, high purity Germanium (HPGe) detectors and the *Visual Gamma* software.

The Appendix contains flow diagrams and the step-by-step instructions for using the Visual Gamma program and specific details of calculations used in the program.

All statements containing the word ‘program’, unless explicitly referring to another computer program, refer to the *Visual Gamma* program.

As *Visual Gamma* is under constant development, changes made to the program will be reflected in updates to this manual – you should ensure that you use the latest version of the manual! Version information is located in the following section (1.3).

The following documents should be read in conjunction with this manual:

ANSI N42.14 (1999) 'American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides'. *eriss* library code STAN ANSI/IEEEE N42.14-1999.

Fox T, Bollhöfer A & Pfitzner J 2011. ***eriss*** gamma spectrometry procedures manual, Internal report (in prep), Supervising Scientist, Darwin.

Knoll GF 1989. Radiation detection and measurement. 2nd edn, John Wiley & Son, New York.

Marten R 1992. Procedures for routine analysis of naturally occurring radionuclides in environmental samples by gamma-ray spectrometry with HPGe detectors. Internal report 76, Supervising Scientist for the Alligator Rivers Region, Canberra. Unpublished paper.

Murray et al 1987. Analysis for naturally occurring radionuclides at environmental concentrations by gamma spectrometry. *Journal of Radioanalytical and Nuclear Chemistry*, Articles 115 (2), 263–288.

Ortec Maestro -32 A65-B32 *Software User's Manual*.

Pfitzner J 2010. ***eriss*** HPGe detector calibration. Internal Report 576, October, Supervising Scientist, Darwin. Unpublished paper.

1.3 Document history, versions

	Date	Version #
Initially written	12–23 March 2007	1.1
Updated	20 September – October 2007	1.2
Updated	February 2008	1.3
Updated	March 2008	1.4
Updated	20 March 2008	1.5
Reviewed and updated (AFB	21–29 July 2008	1.6
Updated (AJE)	13–22 August 2008	1.7
Updated	23 March – 5 April 2010	1.8
Updated (AFB)	7 April – 16 April 2010	1.9
Updated (JLP)	27 July – 6 August 2010	1.95
Updated (AJE)	22 September – 8 October 2010	1.97

2 Introduction

2.1 A brief description of gamma radiation, radionuclides and HPGe detectors

2.1.1 Gamma radiation

Gamma rays are high energy electromagnetic radiation emissions, often called photons. Visible light, ultra-violet light, and x-rays are also radiated energy, listed in increasing energy range. There is an overlap between high energy x-rays and low energy gamma ray energies.

The energy of gamma rays is measured in keV, kilo electron volts and are at the higher end of the frequency scale (Figure 1). 1 keV is equivalent to 1.602 x 10^{-16}J.

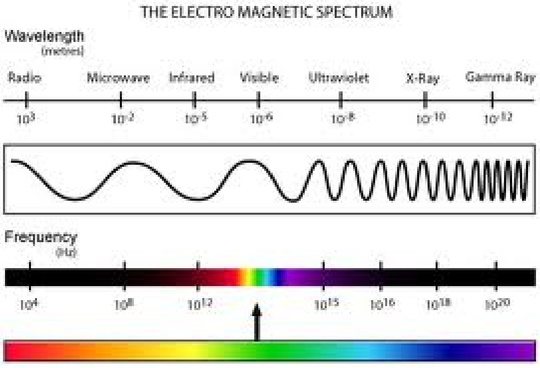
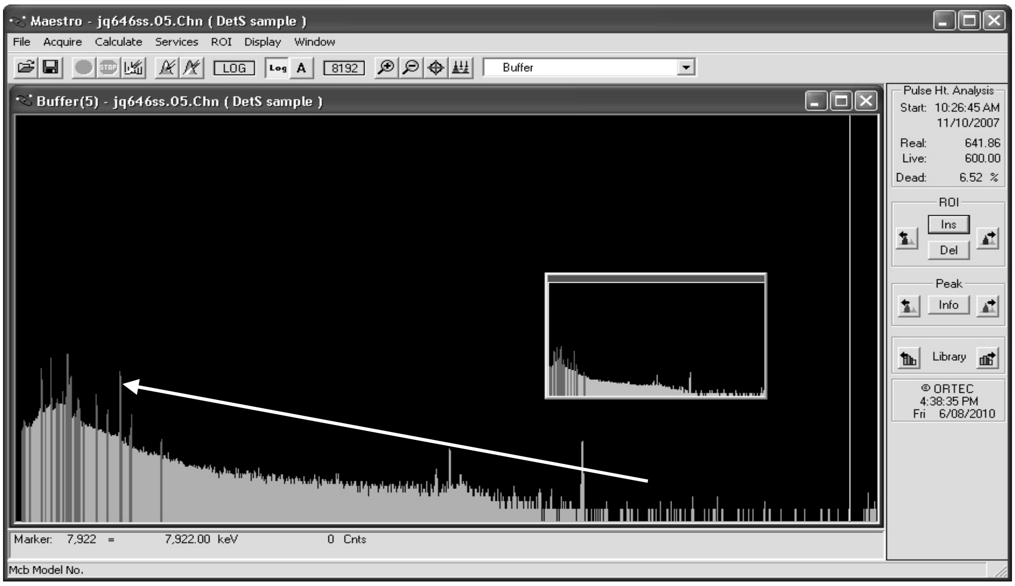


Figure 1 Electromagnetic spectrum with gamma radiation at the higher end of the frequency scale

2.1.2 Gamma ray emitting radionuclides

The stability of radioactive atoms depends on the ratio between neutrons and protons within the nucleus. An atom is unstable when the binding energy is not strong enough to hold the nucleus together. Radionuclides are nuclides that are unstable and undergo radioactive decay. Gamma rays are emitted with every alpha or beta decay, to allow an atom to return to its ground state after a radioactive decay. A gamma ray emitting radionuclide is a radionuclide in an exited state, which emits one or more gamma rays of discrete energies when it returns to its ground state.

The gamma rays are recorded as a distribution of the gamma ray intensity relative to their energy, or a gamma ray spectrum (Figure 2). To analyse a gamma spectrum means to evaluate which radionuclides were present and at what quantity during counting, considering parameters such as sample geometry and counting procedure.


Maestro - jq646ss.05.Chn (DetS sample)

Buffer(5) - jq646ss.05.Chn (DetS sample)

Pulse Ht. Analysis
Start: 10:26:45 AM 11/10/2007
Real: 641.86
Live: 600.00
Dead: 6.52 %

Marker: 7,922 = 7,922.00 keV 0 Cnts

Mcb Model No.

Figure 2 A typical HPGe gamma emission spectrum, with energy channels on the x-axis and number of counts per channel on the y-axis. Counting details are displayed to the right of the spectrum plot. The white arrow shows the trend of increasing background counts at lower energy from the Compton effect.

2.1.3 Principle of HPGe detector operation

High purity Germanium detectors operate in a very low temperature environment to reduce thermally generated electronic noise. These low temperatures are either achieved by cooling with liquid nitrogen (-196ºC) or, more recently, by electrical cooling. At ***eriss***, both liquid nitrogen cooled and electrically cooled detectors are operated.

A vacuum around the detector insulates the cooled components from ambient temperatures. Steel lined lead castles reduce the amount of background radiation reaching the detector.

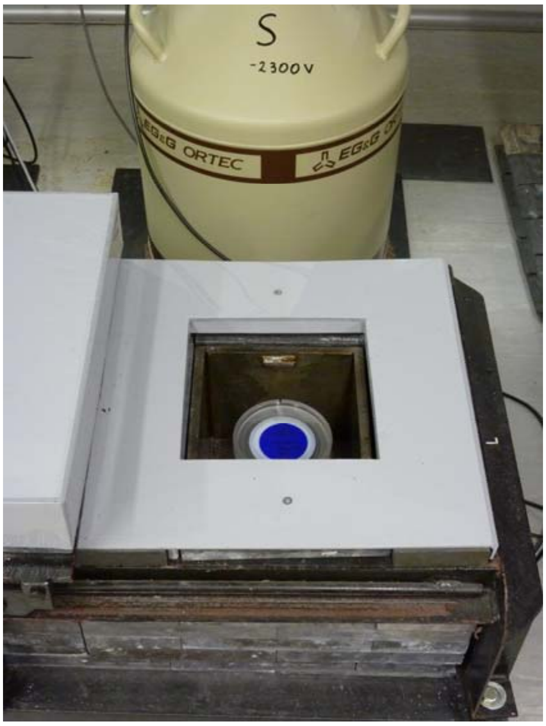


Figure 3 The steel lined lead castle of detector 'S' with lid open, showing the HPGe detector endcap, under the dark blue circle. There is a plastic locating device on top of the detector to maintain consistent positioning of the sample to be counted. The liquid nitrogen dewar is the beige coloured vessel in the background.

Each interaction of a photon with the detector causes a voltage change which is recorded as a count event by the electronic system attached to the detector. The maximum of each voltage fluctuation is a measure of the total photon gamma energy (in keV or kilo electron volts) of that event. These voltages are amplified and then converted to a digital signal by an Analogue to Digital Converter (ADC) and stored in a 'channel' which correspond to that energy. In the case of the eriss HPGe detectors, the photon energy range of 0 – 1,500 keV is divided into 8194 channels that gives a resolution of approximately 0.18 keV/channel.

The gamma rays interact with the detector by 1) Photo-electric, 2) Compton or 3) Pair production effects. In the photo-electric effect, the gamma ray photon, causes an electron to be ejected with an energy equal to the energy of the photon minus its binding energy. As photon energy increases, the photo-electric effect progressively gives way to the Compton and pair-production effects. The Compton effect occurs when the photon energy is not entirely spent in the detector and the resulting electron energy is less than the total of the incident photon. The electron energy E_e measured and recorded is a proportion of the full photon energy and less than a maximum energy characteristic for the gamma energy of the incoming photon.

This maximum energy, the so-called Compton edge, is described via:

$$E_{\max} = \frac{2E^2}{m_e c^2 + 2E}$$

Where:

E: Energy of the incoming photon

m_e: electron mass

c: speed of light

The Compton effect causes an increase in the lower energy background with increasing sample activity (see Figure 1). As a photon energy of greater than 1022 keV is needed to produce an electron/positron pair, this effect is of little concern in environmental samples, however, it does produce the e^+ annihilation peak at 511 keV.

One significant advantage of gamma spectrometry is that there is usually no chemical pre-treatment of the samples. Therefore the gamma spectrometric analytical process is considered ‘non-destructive’. As the material being analysed is not chemically altered, it is possible to conduct chemically analyses on a sample following gamma spectrometric analysis.

2.2 Gamma spectrometry analysis – an overview

Gamma spectrometry analysis measures gamma-rays energies and identifies the radionuclides present in a sample during counting. With given calibration factors it is possible to determine the activity of those radionuclides in Becquerel (Bq) and their activity concentration in Bq per kilogram of sample material. One Bq of activity is defined as one radioactive disintegration per second.

Table 1 Radionuclides and associated gamma energies (in keV) for which Visual Gamma is designed to provide direct gamma spectrometric analysis

^{238}U series	^{232}Th series	Other Isotopes
^{234}Th @ 63 keV	^{228}Ac @ 338 keV	^{40}K @ 1461 keV
^{234}Pa @ 1001 keV	^{228}Ac @ 911 keV	^{137}Cs @ 662keV
^{230}Th @ 68 keV	^{228}Ac @ 969 keV	
^{214}Pb @ 295	^{224}Ra @ 241 keV	
^{214}Pb @ 352 keV	^{212}Pb @ 239 keV	
^{214}Bi @ 609 keV	^{212}Bi @ 40 keV	
^{210}Pb @ 46 keV	^{208}Tl @ 583 keV	

Table 2 Radionuclides indirectly derived from the measured radionuclides in Table 1

^{238}U series	^{232}Th series
^{238}U	^{228}Ra
^{226}Ra	^{228}Th
^{235}U	

The accurate and precise analysis of a HPGe gamma spectrum is only realised after a long and complex process involving all of the following:

Knowledge

To develop an effective gamma spectrometry analytical method, an understanding of:

- The principles of the interaction of gamma emissions with matter,
- radionuclide isotope equilibrium,
- counting statistics,
- the operation of the HPGe detector systems, and
- procedures for spectrum analysis

is required. This knowledge is also useful for the interpretation of gamma spectrometric results.

Instrumentation

Optimum instrument settings are necessary to achieve the best results from all analytical equipment. The HPGe detector manufacturers provide excellent manuals for this purpose (See Canberra Industries and Ortec Instruments manuals for HPGe detectors and associated electronic equipment). ***eriss*** detectors are set up and maintained according to the manufacturer's recommendations.

A measure of detector performance is a lower level of detection and smaller uncertainty in the analytical result. It is therefore essential to reduce the background counts as much as possible and to accomplish this ***eriss*** use steel lined lead castles surrounding the detector to shield the detector from outside radiation, thereby reducing the background counts.

Sample tracking

The implementation of a sample and analysis coding system provides a method of tracking samples within the EnRad laboratory and ensures consistency in analytical procedures. See Appendix H 'Sample and Analysis Coding'.

Sample preparation, pressing and casting

Standardised preparation and analysis methods give results in consistent units for similar materials, e.g. sediment activity concentrations in Bq/kg dry weight. This facilitates direct comparison with results from other laboratories and with published data.

For sample preparation, pressing and casting procedures, see 'Gamma Spectrometry Procedures Manual' (Fox et al 2011).

Details of the pressing or casting are entered in the database via *Visual Gamma*.

Delay for ^{226}Ra equilibrium

A delay of at least 23 days between pressing or casting of the sample and counting on the HPGe detectors is essential for ^{226}Ra and its progeny ^{214}Pb and ^{214}Bi to reach equilibrium so that the progeny can be used to calculate the activity of ^{226}Ra in the sample. To achieve this equilibrium, the 'press' container or 'cast' must be completely gas tight to retain the ^{222}Rn (radon) gas, a decay product of ^{226}Ra.

Counting on the HPGe detector

For most standards and samples, a one day (84 kiloseconds) counting period is sufficient to provide activity concentrations within acceptable limits, see 'Appendix F Minimum

Detectable Activity (or Lowest Limit of Detection)'. *Visual Gamma* provides the interface for starting & stopping counting, and for spectrum storage. The process of counting a sample and saving the spectrum is illustrated in Appendix A.3 'Counting a sample, standard or background and saving the spectrum'

Calibration of the HPGe detector

Rigorous calibration procedures, using traceable standards, ensure the correct calibration factors for the detectors. For further details refer section 4 'Analysing standards and creating or updating calibration'.

Analysis of the spectrum

The principles of analysis are explained in detail in section 6 'Steps in calculating the activities in samples'.

Reporting of results

The results of the above analytical procedures are activity concentrations of radionuclides, typically in units of Bq/kg, which are then reported to internal or external clients or used within EnRad projects.

For detail of this process refer section 7 'Reporting of results'.

Quality assurance (QA) and validation

Frequent counting of backgrounds and stability standards provides evidence that the detectors are operating within specified tolerances. For detail of this process refer section 9 'Quality Assurance'.

3 Features of *Visual Gamma*

3.1 Sample registry

All samples to be analysed, including backgrounds and standards, must first be entered in the Sample Registry program. This ensures that sample details, such as type of sample, sample mass, collection and preparation date, are available for the *Visual Gamma* program during analysis. Counting and subsequent analysis is only enabled when the above details have been entered in the Sample Registry. The Sample Registry is a part of the SQL database.

3.2 ROI analysis method

The analysis method outlined below is significantly different to the Gaussian curve fitting of individual isotope peaks, which is used by most commercially available gamma analysis programs. Over the years various commercial gamma spectrometry analysis programs have been evaluated and it has been found that, for the low radionuclide activity concentrations that are typically found in environmental samples measured at ***eriss***, the ROI method employed here has given more reliable and accurate results than the curve fitting techniques (pers comm R Marten, H Alkansis, C Leslie, A Murray, J Pfitzner). The process of setting these ROIs and subtracting the background is outlined below.

- for all samples default regions of interest (ROIs) for each isotope for the Peak (Pk), Left Background (LBG) and Right Background (RBG) are updated from stability standards which are counted weekly
- a Test Source is counted immediately after each sample count for energy calibration

- interactive energy calibration where the operator ensures that the calibrated ROIs are aligned with the Test Source spectrum
- new ROIs are calculated for each spectrum, based on the shift of the Test Source energy calibration
- for each isotope line, the operator has the ability to visually inspect and adjust the peak and background ROIs to optimise the fit of the ROI to the spectrum
- instrument background calculations use the ROIs generated by a recently counted stability standard to analyse the background spectrum without operator input
- four (4) most recent background counts are averaged for the updated background which is then used in all subsequent analyses
- standardised and referenced formulae for the calculation of weighted averages and all forms of uncertainties.

3.3 ***eriss*** calibration method

Calibration is by the direct measurement of radionuclide standards prepared in the same geometry and matrix as the samples to be analysed. This method has three distinct advantages over other calibration methods in that it eliminates the need to:

- interpolate between measured efficiency values,
- correct for coincidence summing and
- account for the uncertainty in gamma-ray emission probabilities.

The calibration procedures take into account the know interferences of the following isotope pairs at the specified peak energy:

63 keV - ^{234}Th and ^{232}Th series

186 keV - ^{235}U and ^{226}Ra

241 + 242 keV - ^{214}Pb and ^{224}Ra

For further details refer to IR576 (***eriss*** HPGe detector calibration – Pfitzner 2010).

3.4 Uncertainty calculations

The uncertainties on all calibration factors, backgrounds and internal calculations are at 1 Standard Deviation (1 s.d.).

Relative uncertainties are propagated using:

$$\Delta_{total} = ({}_{n}\Sigma\Delta_{n}^{2})^{1/2}$$

with

Δ_{total}	total relative uncertainty
Δ_{n}	relative uncertainty of each contributing step n.

The uncertainty propagation changes when calculating uncertainties on weighted averages, see Appendix C ‘Sources of uncertainty and propagation of uncertainty equations’ and Appendix D ‘Equations for the calculation of weighted average’.

3.5 SQL database

An SQL database stores the following data:

- sample details, such as collection site and date, etc,
- the energy calibration derived ROIs for all isotope lines for each detector, including the continuum background ROIs,
- the instrument backgrounds and uncertainties for each detector,
- calibration factors and uncertainties for each geometry for each detector,
- all sample data calculated during the analysis process each time an analysis is completed, namely:
  - all counts for each channel of the sample spectrum
  - energy calibration shifted ROIs for peaks and backgrounds,
  - actual peak and background ROIs used in the sample analysis,
  - initial peak areas and uncertainties from the counting statistics and continuum background subtraction,
  - peak areas and uncertainties when normalised to time (1 ksec), in count/ksec,
  - peak areas and uncertainties after subtraction of instrument/matrix backgrounds,
  - all values and uncertainties as the program steps through interference corrections
  - the final calculation of activity concentration per mass (Bq/kg).

4 Analysing standards and creating or updating calibration

To achieve good accuracy in gamma spectrometry it is essential that samples be homogenous when presented to the detector. For radionuclide standards this is especially critical because typically a small volume of high activity source material is mixed in a large volume of low activity matrix material. The calibration factors derived from these standards are used to calculate the activity concentrations in many samples. Standards are prepared in (at least) triplicate so that systematic uncertainties of the calibration may be determined.

The calibration data consists of: (1) the calibration factor, (2) the uncertainty from counting statistics, (3) the systematic uncertainty, and (4) the calibration ratios and uncertainties, such as, the 63keV peak compared with the 338, 911 & 969 keV peaks. This ratio provides the ^{232}Th series contribution to the 63keV peak which is the principal ^{234}Th peak used in the calculations of $^{234\text{Th}}$ activity.

Except for ^{226}Ra and ^{232}Th ratios, an accurate value of activity and associated uncertainty of the standards is required for analyses. A determination of systematic uncertainty by R Marten (1992) took into account all possible uncertainties, e.g. those relating to: aliquot of standard, half-life, dilution and weighing, in estimating the total uncertainty of a standard. The derived systematic uncertainties are combined with the counting uncertainty to calculate the final calibration uncertainty for each isotope line. See Appendix C, ‘Sources of uncertainty and propagation of uncertainty equations’.

All calculated uncertainties are 1 standard deviation (1sd) in the same units as their associated value.

4.1 Radium (^{226}Ra)

A pure ^{226}Ra source must be used for this standard, not ^{238}U which contains ^{235}U emitting a gamma-ray at 185.7 keV which causes interference with the 186.0 keV line of ^{226}Ra.

An accurate measure of the activity is not required as only the ratios of the net counts in the 186 keV peak with those peaks at 242, 295, 352 and 609 keV, respectively, are produced from the analysis of this standard. However, the ^{226}Ra standard must have been cast or sealed for at least 23 days to allow the radon daughters (^{214}Pb & ^{214}Bi) to reach equilibrium with radon (^{222}Rn).

The calibration ratios and 1 standard deviation (1sd) uncertainties for the following isotopes are calculated:

- 186 keV / 242 keV
- 186 keV / 295 keV
- 186 keV / 352 keV
- 186 keV / 609 keV

The ratios for each geometry and detector are stored in the database for later use in the ^{238}U calibration.

The process for deriving the calibration factors is described in ***eriss*** HPGe detector calibration, Internal Report 576 (Pfitzner J 2010).

4.2 Uranium (^{238}U) Series

Prerequisite 1

The ratios calculated above from a ^{226}Ra standard in the same geometry and matrix on the same detector will be required to complete the ^{238}U calibration. These ratios are used to calculate the contribution of ^{226}Ra to the 186 keV peak, and thus by subtraction, the ^{235}U counts in this peak. Ensure that the ^{226}Ra ratios used here are from an equivalent matrix and geometry as the ^{238}U standard being analyzed.

Prerequisite 2

It is essential that the ^{238}U standard have been cast or sealed for at least 23 days to allow the radon daughters (^{214}Pb & ^{214}Bi) to reach equilibrium with radon (^{222}Rn). Check that the ^{238}U source material is at least 1 year old which will ensure that ^{234}Th is in equilibrium with its parent, ^{238}U.

An accurate value of the activity of the standard is required, as well as the total propagated uncertainty. ***Check that the activity (in Bq) and total propagated uncertainty are the same as on the preparation card***.

The source isotopes used to make ^{238}U series standards (e.g. Canmet BL-5) have ^{235}U and ^{238}U in their natural activity ratio of ^{235}U/^{238}U = 0.04602. Therefore, after subtraction of the ^{226}Ra contribution at 186keV, an efficiency calibration factor is derived for ^{235}U at 186keV.

The calibration factors for activity and associated 1 standard deviation (1sd) uncertainties in the form of (counts/ksec)/Bq for the following isotopes are calculated:

- ^{235}U @ 186 keV
- ^{234}Th @ 63 keV

- ^{234}Th @ 92 keV
- ^{234}Pa @ 1001 keV
- ^{230}Th @ 67 keV
- ^{226}Ra @ 186 keV
- ^{214}Pb @ 295 keV
- ^{214}Pb @ 352 keV
- ^{214}Bi @ 609 keV
- ^{210}Pb @ 46 keV

The process for deriving the calibration factors is described in ***eriss*** HPGe detector calibration, Internal Report 576 (Pfitzner J 2010).

4.3 Thorium (^{232}Th) Series

The source material used to make ^{232}Th series isotope standards must have all daughter products in secular equilibrium. As the activity concentration of the 1906 $Th(NO_3)_4.4H_2O$ is no longer considered reliable, an IAEA reference material, RGTh-1, is now used as the ^{232}Th standard source. The RGTh-1 however contains ^{238}U, rendering it unsuitable to be used for the 63keV (^{234}Th and ^{232}Th series) ratios. However, the 1906 $Th(NO_3)_4.4H_2O$ can still be used as the source for the derivation of the 63keV series ratios and an additional set of standards of this source are included in the pre-selected counting list.

It is recommended that the ^{232}Th standard source be of at least 50 years age (since initial separation) to ensure that the whole series is in equilibrium with the parent ^{232}Th. Note, that if the ^{232}Th standard is not in equilibrium, the ^{228}Ra, and progeny, will not be of equal activity to ^{232}Th. Alpha spectrometry analysis of the source standard may be required to confirm equilibrium.

Suppliers of certified standard sources should indicate whether or not progeny are in secular equilibrium with ^{232}Th.

An accurate measure of the activity of the standard is required, as well as the total propagated uncertainty.

If the source is free of ^{238}U, then the ratios of the peak at 63 keV to those at 338, 911 & 969 keV are calculated. These ratios provide an estimate of the ^{232}Th series contribution to the peak at 63 keV in samples, and thus by subtraction, the net ^{234}Th (^{238}U series) counts at 63 keV.

The calibration factors for activity and associated 1 standard deviation (1sd) uncertainties in the form of (counts/ksec)/Bq for the following isotopes are calculated:

- ^{228}Ac @ 338 keV
- ^{228}Ac @ 911 keV
- ^{228}Pa @ 969 keV
- ^{224}Ra @ 241 keV
- ^{212}Pb @ 239 keV
- ^{212}Bi @ 40 keV
- ^{208}Bl @ 583 keV

The process for deriving the calibration factors is described in ***eriss*** HPGe detector calibration, Internal Report 576 (Pfitzner J 2010).

4.4 All other radionuclides (^{137}Cs, ^{7}Be, ^{40}K, ^{54}Mn, ^{241}Am)

An accurate measure of the activity of the standard is required, as well as the total propagated uncertainty. The uncertainty of the standard source activity is always specified by the supplier of certified standard source isotopes.

For isotopes with short half-lives, such as ^{137}Cs and ^{7}Be, the activity is determined at the time of counting.

The calibration factors in (counts/ksec)/Bq and associated 1 standard deviation (1sd) uncertainties for the specific radionuclide isotope is calculated.

The process for deriving the calibration factors is described in ***eriss*** HPGe detector calibration, Internal Report 576 (Pfitzner J 2010).

5 Analysing backgrounds

Background spectra are of two distinct types. The most common type is an instrument background, where an empty container or blank resin cast is counted.

These instrument backgrounds are usually counted for three days, eg over a weekend, to achieve reasonable counting statistics for the very low activities in backgrounds.

The other background type is the matrix background for a standard, where the matrix material alone is prepared in the same geometry and container as the standard, to establish the contribution of the matrix to the activity counts of the standard. These backgrounds are also usually counted for 2 or 3 days to achieve reasonable counting statistics.

The initial steps of the analysis of a background spectrum follows a similar path to a sample spectrum. However, as background spectrum are usually of very low activity, it is not expected that there will be peaks of sufficient magnitude to manually adjust the ROIs therefore**,** the program automatically uses the ROIs from the most recently counted stability standard.

Instrument, or matrix, backgrounds and associated uncertainties, in counts per ksec, are recorded for all isotope peak ROIs. The background counts/ksec produced for each peak ROI are the equivalent of a ‘P’ file (refer section 8.1).

If the measured background count for each radionuclide is within the QA limits, the counts/ksec data is averaged with the data for the previous four backgrounds for that geometry. In the *Visual Gamma* database, background measurements are stored and can be retrieved for QA plots.

6 Steps in calculating the activity concentration in samples

The sample and associated test source spectra are read or loaded after measurement.

After calibrating the energy using the 186 and 1001 keV peaks from the test source spectrum, the adjusted ROIs are calculated for the sample spectrum. All isotope lines in the sample spectrum are then displayed & checked visually to ensure the best fit of these ROIs to the peak and backgrounds. Each ROI may be manually adjusted to improve the fit to the spectrum. Finally, the activity concentration for each isotope is calculated and reported.

6.1 Continuum background subtraction

The continuum background is the elevation in background counts mostly due to the compton effect and is higher at lower energies (see Figure 2). The average continuum background is subtracted to give the net area for each isotope peak and associated uncertainties - the formulas are published in the Canberra Products Catalogue Ed12 p25, and ANSI N42.14-1999, p62-63. For details refer Appendix E. ‘Peak areas and uncertainties by end point averaging’.

Peak area data and associated uncertainties are available in the ‘P’files (section 8.1) , with all isotope ROIs used in the analysis.

6.2 Normalisation processes

The peak area for a region of interest needs to be normalized to counts/ksec. At this stage peak area and uncertainty are total counts, therefore these must first be divided by the count time (in ksec).

6.3 Instrument/matrix background subtraction

This background is derived from any activity not coming from the source and is minimized by lead shielding around the detector and the sample (see Figure 3). The counts for the four most recent background spectra for that detector are energy aligned using the ROI’s from the counting of a recent stability standard, normalised to 1 ksec and then averaged. This instrument/matrix background is subtracted from the sample spectrum.

6.4 Activity concentration for isotope lines without interferences

1. If the net peak area/ksec is zero or negative, a flag is set to report minimum detection limit. Refer Appendix F, ‘Minimum detectable activity (or lowest limit of detection)’
2. The intensity in cts per ksec is converted into activity, by multiplying net peak area/ksec by the calibration factor (Bq·ksec/kg).
3. The activity is divided by the sample weight to give activity concentration (Bq/kg).
4. Uncertainties are propagated according to the formula found in Appendix C, ‘Sources of uncertainty and propagation of uncertainty equations’.

6.5 Activity concentration for isotope lines with interferences

The following isotope activities cannot be calculated directly from the number of counts in a particilar ROI because the number of counts are derived from more than one isotope.

6.5.1 Thorium 63 keV line (^{234}Th)

To correct for contributions from isotopes in the ^{232}Th series to the 63 keV line of ^{234}Th (^{238}U series), the ratios of 63 keV to 338, 911 & 969 keV as recorded in the calibration file (or database) are used to determine 3 correction factors:

Correction factor (338) = (ratio of 63/338 from cal file) (sample 338 keV net peak area/ksec)*

Correction factor (911) = (ratio of 63/911 from cal file) (sample 911 keV net peak area/ksec)*

Correction factor (969) = (ratio of 63/969 from cal file) (sample 969 keV net peak area/ksec)*

The weighted average of these 3 correction factors is calculated. See Appendix D, ‘Equations for the calculation of weighted average’.

This weighted average of the ^{232}Th contribution is subtracted from the sample net peak area/ksec at 63 keV to give the net ^{234}Th peak area/ksec. A new uncertainty is calculated as the square root of the sum of the squared uncertainties.

6.5.2 Radium 241 keV line (^{224}Ra)

The contribution of the ^{214}Pb 242 keV line is subtracted from the overlapping ^{224}Ra 241 keV. Three high efficiency lines provide alternative values for ^{214}Pb, they are the 295 and 352 keV line of ^{214}Pb and the 609 keV line of ^{214}Bi.

The first step uses the calibration file data to find the ratios, and uncertainties, of the 242 keV line to each of the other three lines. Each ratio is multiplied by its respective net peak area/ksec to give 3 values for the ^{214}Pb contribution to the 241+242 peak in the sample.

Correction factor (295) = (ratio of 242/295 from cal file) (sample 295 keV net peak area/ksec)*

Correction factor (352) = (ratio of 242/352 from cal file) (sample 352 keV net peak area/ksec)*

Correction factor (609) = (ratio of 242/609 from cal file) (sample 609keV net peak area/ksec)*

The weighted average of the three values and associated uncertainty are calculated and this value is subtracted from the 241+242 net peak area/ksec to give the ^{224}Ra 241 keV net peak area/ksec with a new uncertainty.

6.5.3 Uranium 186 keV line (^{235}U)

If the sample was sealed for more than 23 days before counting, ^{222}Rn progeny is used to calculate ^{226}Ra, and thus ^{226}Ra contribution to the 186 keV peak to find ^{235}U net peak area/ksec at this energy. Again, the 295, 352 and 609 keV lines are used. The ratio of the 186 keV line to each of these lines has previously been determined with a pure ^{226}Ra standard. Using this ratio and the measured net counts in the peaks at 295, 352 and 609 keV, the contribution of ^{226}Ra to the 186 keV line and a weighted average of the three results is calculated, to find the ^{226}Ra contribution to the 186 keV peak in the sample.

Correction factor (295) = (ratio of 186/295 from cal file) (sample 295 keV net peak area/ksec)*

Correction factor (352) = (ratio of 186/352 from cal file) (sample 352 keV net peak area/ksec)*

Correction factor (609) = (ratio of 186/609 from cal file) (sample 609 keV net peak area/ksec)*

The ^{226}Ra contribution is subtracted from the total 186 keV net peak area/ksec to give ^{235}U net peak area/ksec at 186 keV after which a value for ^{238}U is calculated (assuming natural activity ratio of ^{235}U:^{238}U= 0.04602). This ^{238}U value may be used to confirm equilibrium between ^{238}U and ^{234}Th, however, the uncertainties may be large and therefore the result of the comparison inconclusive.

Now that all of the interferences have been resolved, each of these isotope lines is multiplied by their calibration factor from the calibration file and divided by the mass of sample to give final activity concentrations in Bq/kg.

7 Reporting of results

Under normal conditions, the sample will have been stored in a radon tight container for > 23 days allowing the following progeny to reach equilibrium with their parents, ^{222}Rn progeny (^{214}Pb, ^{214}Bi) with ^{226}Ra; ^{212}Pb & ^{224}Ra with ^{228}Th, and ^{228}Ac with ^{228}Ra. This means that the parents can be reported, as well as the individual isotopes.

If the sample was counted > 120 days after the sample was sealed then ^{234}Th will be in equilibrium with its parent ^{238}U.

Depending upon the time since casting or sealing the sample container, the following sample equilibrium rules apply to reporting:

If < 2 days, then the following equilibrium conditions apply;

^{228}Ac not in equilibrium with ^{228}Ra, so ^{228}Ra cannot be reported, only ^{228}Ac.

^{212}Pb & ^{212}Bi not in equilibrium with ^{224}Ra. Report as ^{212}Pb & ^{212}Bi.

^{224}Ra not in equilibrium with ^{228}Th. Report as ^{224}Ra.

^{214}Pb & ^{214}Bi not in equilibrium with ^{226}Ra. Report ^{212}Pb & ^{212}Bi as ^{222}Rn.

^{234}Th not in equilibrium with ^{238}U. Report as ^{234}Th.

If between 2 and 4 days, then

^{228}Ac will have reached equilibrium with ^{228}Ra. Report ^{228}Ac as ^{228}Ra.

^{212}Pb & ^{212}Bi will have reached equilibrium with ^{224}Ra. Report ^{212}Pb & ^{212}Bi as ^{224}Ra.

^{224}Ra not in equilibrium with ^{228}Th. Report as ^{224}Ra.

^{214}Pb & ^{214}Bi not in equilibrium with ^{226}Ra. Report ^{212}Pb & ^{212}Bi as ^{222}Rn.

^{234}Th not in equilibrium with ^{238}U. Report as ^{234}Th.

If > 23 days, then

^{228}Ac will have reached equilibrium with ^{228}Ra. Report ^{228}Ac as ^{228}Ra.

^{212}Pb & ^{212}Bi will have reached equilibrium with ^{224}Ra. Report ^{212}Pb & ^{212}Bi as ^{228}Th.

^{224}Ra will have reached equilibrium with ^{228}Th. Report ^{224}Ra as ^{228}Th.

^{214}Pb & ^{214}Bi will have reached equilibrium with ^{226}Ra. Report ^{214}Pb & ^{214}Bi as ^{226}Ra.

^{234}Th not in equilibrium with ^{238}U. Report as ^{234}Th.

If > 120 days, then at last

^{234}Th will have reached equilibrium with ^{238}U. Report ^{234}Th as ^{238}U.

8 Documents produced during analysis

Two documents are produced following the analysis process:

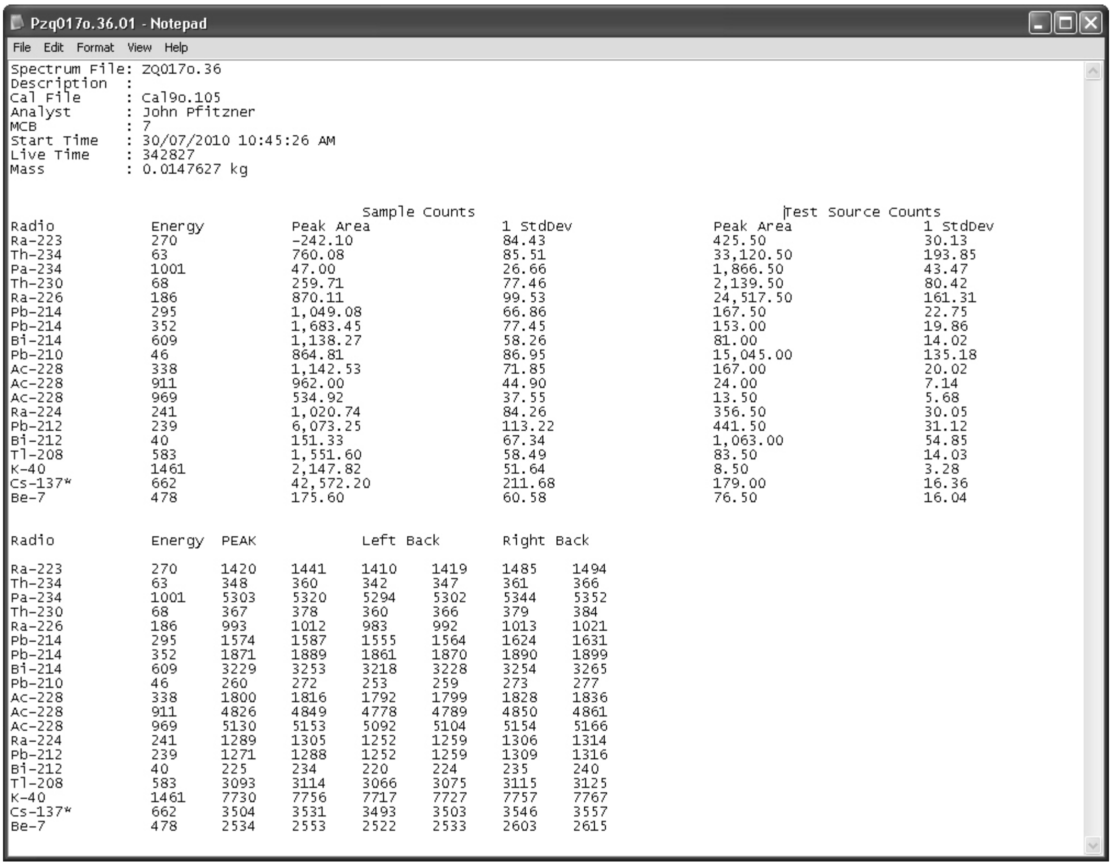
- ‘P’ file
- ‘R file

8.1 ‘P’ files

These files are an emulation of the P (**peak**) type file created by the previous analysis programs.

At the top of the file or more correctly, database subset, are the sample name and description, calibration file, analyst, count date, live time and sample mass. In the main body of the file, the isotopes and gamma emission energies are listed, with net peak areas and 1 s.d. uncertainties (only continuum background subtracted) under the ‘Sample Counts’ heading, and Test Source Counts and 1 s.d. uncertainties. The lower portion displays the final ROI’s channels for peak and left and right backgrounds.

The lower portion displays the current ROIs for the peak channels and the left and right continuous backgrounds. Figure 4 shows an example of the output created by the Visual Gamma program.



```
Spectrum File: ZQ017o.36
Description  :
Cal File     : Cal9o.105
Analyst      : John Pfitzner
MCB          : 7
Start Time   : 30/07/2010 10:45:26 AM
Live Time    : 342827
Mass         : 0.0147627 kg

                                        Sample Counts                                   Test Source Counts
Radio           Energy          Peak Area               1 StdDev                Peak Area               1 StdDev
Ra-223          270             -242.10                 84.43                   425.50                  30.13
Th-234          63              760.08                  85.51                   33,120.50               193.85
Pa-234          1001            47.00                   26.66                   1,866.50                43.47
Th-230          68              259.71                  77.46                   2,139.50                80.42
Ra-226          186             870.11                  99.53                   24,517.50               161.31
Pb-214          295             1,049.08                66.86                   167.50                  22.75
Pb-214          352             1,683.45                77.45                   153.00                  19.86
Bi-214          609             1,138.27                58.26                   81.00                   14.02
Pb-210          46              864.81                  86.95                   15,045.00               135.18
Ac-228          338             1,142.53                71.85                   167.00                  20.02
Ac-228          911             962.00                  44.90                   24.00                   7.14
Ac-228          969             534.92                  37.55                   13.50                   5.68
Ra-224          241             1,020.74                84.26                   356.50                  30.05
Pb-212          239             6,073.25                113.22                  441.50                  31.12
Bi-212          40              151.33                  67.34                   1,063.00                54.85
Tl-208          583             1,551.60                58.49                   83.50                   14.03
K-40            1461            2,147.82                51.64                   8.50                    3.28
Cs-137*         662             42,572.20               211.68                  179.00                  16.36
Be-7            478             175.60                  60.58                   76.50                   16.04


Radio           Energy  PEAK            Left Back       Right Back

Ra-223          270     1420    1441    1410    1419    1485    1494
Th-234          63      348     360     342     347     361     366
Pa-234          1001    5303    5320    5294    5302    5344    5352
Th-230          68      367     378     360     366     379     384
Ra-226          186     993     1012    983     992     1013    1021
Pb-214          295     1574    1587    1555    1564    1624    1631
Pb-214          352     1871    1889    1861    1870    1890    1899
Bi-214          609     3229    3253    3218    3228    3254    3265
Pb-210          46      260     272     253     259     273     277
Ac-228          338     1800    1816    1792    1799    1828    1836
Ac-228          911     4826    4849    4778    4789    4850    4861
Ac-228          969     5130    5153    5092    5104    5154    5166
Ra-224          241     1289    1305    1252    1259    1306    1314
Pb-212          239     1271    1288    1252    1259    1309    1316
Bi-212          40      225     234     220     224     235     240
Tl-208          583     3093    3114    3066    3075    3115    3125
K-40            1461    7730    7756    7717    7727    7757    7767
Cs-137*         662     3504    3531    3493    3503    3546    3557
Be-7            478     2534    2553    2522    2533    2603    2615
```

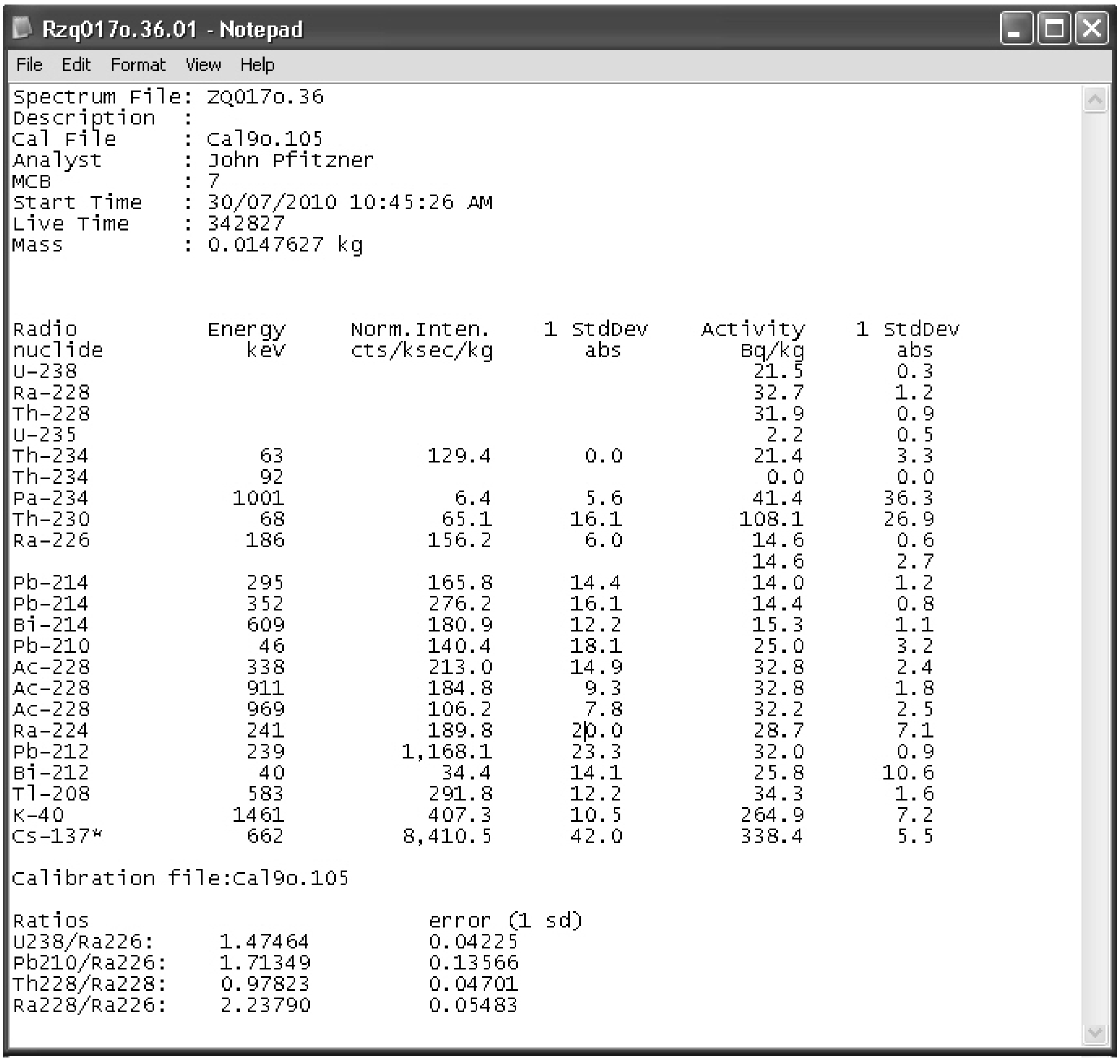
Figure 4 ‘P’file output from Visual Gamma

8.2 ‘R’ files

These files are an emulation of the R (**results**) type files created by the previous analysis programs.

At the top of the file or more correctly, database subset, are the sample name and description, calibration file, analyst, count date, live time and sample mass. In the main body of the file, the isotopes and gamma emission energies are listed, followed by the normalised intensity and the activity concentration, both with 1s.d. uncertainties. Under this dataset, are the ratios of the major radionuclides, with 1.s.d. uncertainty.

Figure 5 shows an examples of an ‘R' type file created by the Visual Gamma program.



```
Spectrum File: ZQ017o.36
Description  :
Cal File     : Cal9o.105
Analyst      : John Pfitzner
MCB          : 7
Start Time   : 30/07/2010 10:45:26 AM
Live Time    : 342827
Mass         : 0.0147627 kg



Radio          Energy     Norm.Inten.    1 StdDev    Activity    1 StdDev
nuclide           keV     cts/ksec/kg       abs         Bq/kg       abs
U-238                                                    21.5       0.3
Ra-228                                                   32.7       1.2
Th-228                                                   31.9       0.9
U-235                                                     2.2       0.5
Th-234             63           129.4       0.0          21.4       3.3
Th-234             92                                     0.0       0.0
Pa-234           1001             6.4       5.6          41.4      36.3
Th-230             68            65.1      16.1         108.1      26.9
Ra-226            186           156.2       6.0          14.6       0.6
                                                         14.6       2.7
Pb-214            295           165.8      14.4          14.0       1.2
Pb-214            352           276.2      16.1          14.4       0.8
Bi-214            609           180.9      12.2          15.3       1.1
Pb-210             46           140.4      18.1          25.0       3.2
Ac-228            338           213.0      14.9          32.8       2.4
Ac-228            911           184.8       9.3          32.8       1.8
Ac-228            969           106.2       7.8          32.2       2.5
Ra-224            241           189.8      20.0          28.7       7.1
Pb-212            239         1,168.1      23.3          32.0       0.9
Bi-212             40            34.4      14.1          25.8      10.6
Tl-208            583           291.8      12.2          34.3       1.6
K-40             1461           407.3      10.5         264.9       7.2
Cs-137*           662         8,410.5      42.0         338.4       5.5

Calibration file:Cal9o.105

Ratios                          error (1 sd)
U238/Ra226:     1.47464         0.04225
Pb210/Ra226:    1.71349         0.13566
Th228/Ra228:    0.97823         0.04701
Ra228/Ra226:    2.23790         0.05483
```

Figure 5 ‘R’file output from Visual Gamma

Using the Visual Gamma database, it is also possible to retrieve results for a group of samples from a project and print all the results into an excel spreadsheet (see ‘Visual Gamma – ***eriss*** Gamma Analysis Operational Manual’, Esparon & Pfitzner, in prep).

9 Quality assurance

The following parameters are logged and can be displayed as plots for quality assurance:

1 Background, from the database of backgrounds spectra, the trend is plotted against time for each detector for all lines.

2 Stability checks, from the database of activity concentration results for each stability count, the trend is plotted against time for each detector for these isotope lines 46.5, 63, 186, 239, 352, 609, 911 & 969 keV.

3 Peak shift, using the Test Source data, the energy drift is plotted against time.

Frequent counting of backgrounds and stability standards provides evidence that the detectors are operating within specified tolerances.

9.1 Stability check standards

Standards have been prepared in Q geometry (~15g of material in Perspex container) to provide a fast (3 hour count) method of checking the accuracy of the following parameters:

1 mechanical stability, eg temperature of germanium crystal, integrity of vacuum seals, any physical damage to the window or cryostat,

2 electrical and electronic stability, eg spectrum shift and drift, electronically noisy components, main power supply surges and sags, accidental changes to instrument module settings, any physical damage to cables or connections and

3 analysis program function, eg alignment of ROIs, calculation of background and peak net areas, conversion to activity concentration, estimation of uncertainty, determination of attenuation and decay correction factors and reporting of results.

The source materials for these standards are Canmet BL-5 for the ^{235}U series and a calibrated solution of 1906 thorium nitrate for the ^{232}Th series. Activities for each standard were calculated to give around 20,000 net counts in most of the isotope peaks in both radionuclide series. This is equivalent to 0.6 g of BL-5 and 2 ml of the 200Bq/ml thorium solution in each standard. These quantities were added to 15g of finely ground Flying Fox sand.

10 Future developments

NOTE: Attenuation correction and Decay Correction have not yet been implemented.

It has been recognised that the following automated features outlined below would further enhance the functionality of the software. These functions were previously done either by separate programs or by manually entering the data into spreadsheets.

10.1 Attenuation correction

NOTE: Attenuation correction has not yet been implemented.

After the calculation of activity, attenuation is always the next correction to be applied – if decay or other corrections are to be done, they always follow the attenuation correction. Attenuation correction should not be applied after decay correction.

The Test Source count that is included with each sample can be used for a rapid and accurate method of attenuation correction. If all of the radionuclide standards for a particular geometry are made with one matrix, eg Flying Fox sand, then this will simplify the calculation of the attenuation correction for that geometry.

The Test Source provides attenuation correction factors for 46.5, 63 and 186 keV.

The following steps are prerequisites for the calculation and application of the attenuation correction factor:

1 Ensure that there is a background matrix standard available consisting of only the standard matrix material, eg Flying Fox sand, in the same geometry and weight as the radionuclide standards.

2 Establish a good background value (in counts/ksec) at 46.5, 63 & 186 keV ROIs from a 2 day count of the standard matrix (eg only Flying Fox sand).

3 Measure the Test Source transmission at 46.5, 63 & 186 keV ROIs through the background matrix over 600 seconds (in counts/ksec), to allow a direct comparison with

4 Calculate similar transmission values for the 46.5, 63 and 186 keV lines for the Test Source counted with the sample (in counts/ksec), however, in this case the sample activity (in counts/ksec) must be subtracted as the background from the Test Source count. This net transmission value is the Sample Test Source Transmission (Sample TST).

5 The following formula is used at each isotope energy to give an attenuation corrected activity:

$$A_{Attenuation\,Corrected} = A_{Measured} * \left(\frac{(\text{Standard}\,TST)}{(\text{Sample}\,TST)} \right)$$

When attenuation correction factors are not available for a particular isotope energy, due to the absence of a Test Source peak at that energy (eg ^{241}Am @ 59keV), attenuation correction factors are interpolated from an attenuation curve for that sample derived from the available Test Source energies either side of the energy of interest.

Attenuation correction factors have been checked for geometry Q by making standards with accurately know amounts of BL-5 added to various matrix materials which have different attenuation to the Flying Fox sand. These standards have been counted and their activity calculated with and without attenuation correction to confirm that the attenuation correction factors are valid.

The attenuation correction will vary considerably with energy, most attenuation at lower energies, much less at higher energies.

Warning: the smallest container size may require a smaller Test Source to account for the very small area of the standard and sample material compared to the wider distribution of the radionuclides in the Test Source.

Refer to the following papers for a more detailed description of the process:

Cutshall NH, IL Larsen & CB Olsen 1983. Direct analysis of Pb-210 in sediments: self absorption corrections. *Nuclear Instruments & Methods* 206, 309–312.

Larsen IL, Cutshall NH & Olsen CB 1976. Nondestructive low-energy photon analysis of environmental samples. In *Proceedings of the ERDA Symposium on x- and gamma-ray sources and applications*. ed Vogt JR, University of Michigan, Ann Arbor, CONF-760539, 206–209.

10.2 Decay or ingrowth correction to time of collection

If the activity of any short lived isotope is required to be expressed as activity at the time of collection, it is most important that an initial count of the sample be made as soon as possible after collection.

There are two modes of correction:

1 where *independent* short lived isotopes are decaying

2 where short lived isotopes are out of equilibrium with their long lived parents

For isotopes of mode 1, ^{7}Be ($t_{1/2}$ = 53.3 d) and ^{137}Cs ($t_{1/2}$ = 30.2 y), the correction for decay which has occurred during the time since sample collection is the usual form of

$A_{corr} = A_{measured} \cdot e^{\lambda t}$

Where:

A_{corr}: corrected activity to time of sample collection,

$A_{measured}$: the activity at the time of counting

e: natural log (2.71878)

λ: decay constant for isotope, and

t: time during which decay occurred (in same units as used in calculating the decay constant).

For the mode 2 type isotopes such as ^{234}Th ($t_{1/2}$ = 24.1 d) and ^{210}Pb ($t_{1/2}$ = 22.6 y), the decay correction is not so simple. A decay, or ingrowth, correction is only required if these isotope are out of equilibrium with their parents ^{238}U and ^{226}Ra, respectively.

Either an accurate knowledge of the ^{238}U activity is required to correct for ^{234}Th decay/ingrowth since collection time to first counting, or a second count is required to find the amount of ^{234}Th decay or ingrowth. If ^{234}Th – ^{238}U activity is negative, then the ^{234}Th is growing in to reach equilibrium with the ^{238}U. Equilibrium between ^{238}U and ^{234}Th is only assumed after 5 half lives of ^{234}Th, ie about 6 months from time of collection.

Similarly for ^{210}Pb, a knowledge of ^{226}Ra is required, to allow correction for the ‘unsupported’ ^{210}Pb. Equilibrium between ^{226}Ra and ^{210}Pb is only assumed after 5 half lives of ^{210}Pb, ie about 113 years from time of collection.

In both mode 2 cases the preferred method is to subtract the parent radionuclide (^{238}U or ^{226}Ra) from the isotope (^{234}Th or ^{210}Pb, respectively) to allow correction to be made only on that portion of the progeny which is not in equilibrium with the parent.

11 Warnings and caveats

To obtain the best results from the Ge detectors and from this program, it is the responsibility of the user to ensure that good laboratory practices are followed. For example, samples must be homogenous and carefully prepared to avoid contamination, unscheduled dilution and radon escape. The detector maintenance and operating procedures must be performed diligently according to the standard procedures. The sample preparation and counting environments must be kept clean to avoid contamination and increased background activity, Any problems with the detector, the associated electronic instrumentation or with the *Visual Gamma* program must be immediately passed on to the Program Leader of the Environmental Radioactivity Program for inspection and repair or correction.

12 Glossary

Accuracy – a measure of how close a result or set of results is to the true value and is related to the systematic error. See also ‘Random Error’, ‘Systematic Error’ and ‘Precision’.

Activity – quantities of radioactive materials are expressed in a form which relates to the decays per unit time of a particular radionuclide, or activity. One Becquerel (Bq) is one disintegration per second, one Curie (Ci) is $3.7 \cdot 10^{10}$ disintegrations per second (or $3.7 \cdot 10^{10}$ Bq). *eriss* activity concentration are expressed in S.I. units as Bq/kg or mBq/L.

Alpha decay – a highly ionising form of particle radiation. It consists of two protons and two neutrons bound together into a particle identical to a helium nucleus; hence, it can be written as He^{2+}.

Alpha particles - are emitted by radioactive nuclei such as uranium or radium. This sometimes leaves the nucleus in an excited state, with the emission of a gamma radiation removing the excess energy. In contrast to beta decay, alpha decay is mediated by the strong nuclear force. Classically, alpha particles do not have enough energy to escape the potential of the nucleus. However, the quantum tunnelling effect allows them to escape.

When an alpha particle is emitted, the atomic mass of an element goes down by roughly 4 atomic mass units (amu), due to the loss of 4 nucleons. The atomic number of the atom goes down by 2, as the atom loses 2 protons, becoming a new element. An example of this is when radium ($_{88}Ra$) becomes radon ($_{86}Rn$) gas due to alpha decay.

Amplifiers – electronic devices used to magnify the weak electrical signal received from the germanium crystal before analogue to digital conversion. For gamma spectrometry work amplifier requirements include highly stable and linear performance.

Analogue to digital conversion (A-D) - analogue to digital converters (ADCs) measures the amplified energy pulse and places a ‘count’ in an appropriate digital energy bin or channel. The energy range from 0 to ~1500keV is divided into 8192 (2^{12}) channels. The detector response, amplification and conversion of analogue to digital is close to linear, allowing for relatively easy identification of specific isotope peaks. A plot of energy vs channel number is interesting, but not an essential part of a QA scheme.

Attenuation – the net loss at the detector of primary photons of a given energy resulting from their interaction with matter, either due to scattering or absorbance within the sample matrix or in the material between the sample and the detector crystal.

Background

Continuum Background is the spectrum region around a peak which provides a measure of the average Compton continuum contribution to the counts in the peak ROI. The continuum background increases markedly with higher radionuclide activity, and increases slightly with increasing detector size. However, the peak-to-Compton ratio (p/C) increases for larger detectors, which means that the ratio of the signal in the photo-peak to the Compton background counts will improve.

Instrument or Detector Background is the background above the continuum background from the spectrum of an empty sample container on the detector, expressed as a number of counts/ksec for a peak ROI. Main background contributors are the nearby ground, building materials and high energy cosmic radiation. Detector materials also contribute to the background and all manufacturers have special Low Background models.

Matrix Background is the background (counts/ksec) above the continuum background from the spectrum of a material used to make a standard, e.g. Flying Fox sand, expressed as a number of counts/ksec for a peak ROI.

Becquerel (Bq) – a measure of radioactivity, disintegration per second.

Beta Decay – is a type of radioactive decay in which a beta particle (an electron or a positron) is emitted. In the case of electron emission, it is referred to as ‘beta minus’ (β^-), while in the case of a positron emission as ‘beta plus’ (β^+). In β^- decay, the weak interaction converts a neutron into a proton while emitting an electron and an anti-neutrino. In β^+ decay, a proton is converted into a neutron, a positron and a neutrino. The resulting radionuclide usually is in an excited state, and decays via the emission of a photon.

Calibrating a detector

1 Energy calibration: calculate the energy per channel (8192 channels) + offset,

2 Efficiency calibration: to determine the conversion factor for each isotope (efficiency),

3 Background calibration: to determine the background for each isotope of interest.

See also ‘Background’, ‘Conversion factors’ & ‘Standards and source materials’.

Channel – the energy range (0 to 1500 keV) is divided into 8192 separate channels.

Compton background – when an incident gamma photon interacts with an internal layer electron, the electron is ejected with a certain speed and the incident photon is scattered with a reduced energy. In the process the energy and momentum are conserved. If the scattered photon leaves the detector, the resulting energy will be smaller than in the case of complete absorption, and a lower energy count will be registered in the spectrum. As these reduced energy photons accumulate, they increase background at the lower energy region of the spectrum.

Conversion factors – a number which converts values from one form to another, e.g. from counts/ksec to Bq/kg using conversion factor of (counts/ksec)/Bq and knowledge of weight. All conversion factors should be properly identified and the equation for the implementation of the factor must be stored with the converted data.

Cryogenic cooling – is necessary to reduce thermal noise in the germanium crystal. See Liquid Nitrogen Cooling and Electrical Cooling.

Daughter products (Progeny) – a synonym for the product of a radionuclide decay event. These progeny can be either stable or radioactive.

Decay series (or decay chains) – a group of radionuclides related by parent or progeny to all other radionuclides in the series, e.g. Uranium (^{238}U).

Detector area – refers to the end area of the germanium crystal cylinder. These range from about 1000 mm^2 to 4000 mm^2.

Detector end cap – is the outer container of the detector assembly. The important characteristics of this end cap are that it should be light and vacuum tight and allow the maximum amount of gamma radiation to pass through to the detector. ^{210}Pb is of major interest to many users and this isotope has a relatively low energy photon of 46.5 keV. Detector specifications usually state a minimum attenuation for 40 keV. Various materials are used for detector end caps, aluminium, magnesium, copper, beryllium and carbon fibre composite for example.

Electrical cooling – compressed refrigerant electrical cooling of germanium detectors has been available for over 10 years. However, recent developments in the use of Stirling pumps and helium refrigerant have made the cooling of detectors without liquid nitrogen a viable option. Although the colder temperature available with liquid nitrogen gives slightly better performance, measured in better resolution, the occupational health and safety advantage of electrically cooled detectors far outweighs any slight performance edge of liquid nitrogen cooling. Electrical cooling is also cheaper, due to the high price of liquid nitrogen in Darwin.

Efficiency vs wnergy – the larger germanium crystals increase the Relative Efficiency, but it is not a linear increase for all energies. Small crystals are able to count nearly all low energy photons but are not effective for high energy photons. In the case of the high energy photon and the small crystal, only a part of the photon energy may be spent in the crystal before it exits with some energy remaining. The photon is seen by the detector system as a photon of lower energy and appears on the lower energy region of the spectrum. This randomly contributes to a higher low energy background (the continuum or Compton Background). The photon energy can fall in another isotope emission peak energy region where it will appear as a photon of that lower energy isotope emission. Larger crystals are more effective in completely trapping more of the higher energy photons, but not much better at the low energy photons. Large crystals usually exhibit improved low energy continuum background.

Equilibrium – a radionuclide is in secular (natural) equilibrium when the activity of the progeny is equal to that of the parent. Put simply, equilibrium is the equal activity concentrations between radionuclides within the same series.

Error – Error is defined as the difference between an individual result and the 'true' value of a measurand and can be applied as a correction to the result (whereas uncertainty takes the form of a range which may be applied to all similar determinations so described, and cannot be used to correct a measured result). There are two components of errors, Random errors, or unpredictable influences, and systematic errors, which vary in a constant or predictable way. Results should be corrected for systematic errors. Spurious errors, such as instrument malfunction or failure to subtract background, should be confirmed and the results rejected if exact correction is not available. See also 'Systematic Error'.

FWHM – Full width at half maximum**.** The width of the peak at half of the maximum peak height with the continuum background removed. A measure of energy resolution.

Gamma radiation – an energetic form of electromagnetic radiation produced by radioactive decay or other nuclear or subatomic processes such as electron-positron annihilation. Gamma rays form the highest-energy end of the electromagnetic spectrum. They are often defined to begin at an energy of 10 keV, corresponding to a minimum frequency of 2.42 EHz, or a maximum wavelength of 124 pm, although electromagnetic radiation from around 10 keV to several hundred keV is also referred to as hard X-rays. It is important to note that there is no physical difference between gamma rays and X-rays of the same energy – they are two names for the same electromagnetic radiation. Rather, gamma rays are distinguished from X-rays by their origin. Gamma radiation is produced by nuclear transitions, while X-rays are produced by transitions of electrons in the atom shell from an excited state (or states) or energy transitions due to accelerating electrons. Because it is possible for some electron transitions to be of higher energy than some nuclear transitions, there is an overlap between what we call low energy gamma rays and high energy X-rays.

Gamma radiation is a form of ionising radiation; more penetrating than either alpha or beta radiation (neither of which is electromagnetic radiation), but less ionising. For instance, gamma radiation will pass through 1 cm of aluminium, while an alpha ray will be stopped by even a single sheet of paper.

Germanium detector – is a semiconductor diode made of hyper pure germanium in which the germanium is sensitive to ionising radiation, particularly X-rays and gamma rays (photons). An electric field is established across the germanium crystal (also called the intrinsic or depleted region).

Half-life – the time required for one half the atoms in a radioactive substance to decay.

J style cryostat – refers to the configuration of the liquid nitrogen dewar and the detector head. The eriss detectors are all housed in J style cryostats. Other arrangements are where the detector sits atop the dewar or faces out from the base of the dewar. Dewars also come in different sizes, from 2 to 50 litres, and as portable and all-orientation styles, for specialised applications. See also ‘Liquid Nitrogen Cooling’ & ‘Temperature Cycling’. Similar configurations are available for electrically cooled detector assemblies.

keV – kilo electron volts, units of gamma radiation energy.

Liquid nitrogen cooling – is necessary to lower the thermally generated background in the germanium crystal and initial (FET) amplifier. Nitrogen is liquid below -196° C. Alternative cooling via electrically powered Stirling pumps is now suitable as a long-term replacement for liquid nitrogen which is potentially a serious asphyxiant hazard.

Live time – the time interval of a count during which the counting system is capable of registering counts. Elapsed, or real, time equals live time plus dead time.

Matrix – the composition of material that makes up a standard or a sample.

MCA – multichannel analyser, an electronic device that records and stores pulses according to their height. The three functional elements are, 1) an ADC to measure pulse height amplitude, 2) memory registers to tally pulses within given amplitude increments and 3) an input/output section that permits transfer of accumulated spectral information to other devices, or storage media.

Parent radionuclide – the radionuclide from which a new isotope (progeny) is formed.

Passive shielding – usually consists of lead and steel and or copper which surrounds the detector and sample and serves to lower the background contribution from the ground, building materials and from cosmic radiation. Relatively cheap and reasonably effective if the shielding materials are of low activity themselves.

Peak – the region in a spectrum where an accumulation of emissions of one energy look like a mountain peak when plotted against energy. See also ‘Region Of Interest’ or ROI. The peak ROIs are highlighted with different colours on computer displays of a spectrum.

Precision – the measure of how much variation there is within a set of results of the same sample or standard. This is related to the random error. See also ‘Random Error’, ‘Systematic Error’ and ‘Accuracy’.

Progeny – a synonym for the product of a radionuclide decay event. These progeny can be either stable or radioactive.

Random error (or random uncertainty) – is produced by unknown and unpredictable variations in the analysis situation. See also ‘Precision’, ‘Systematic Error’ and ‘Accuracy’.

Region of interest (ROI) – is a series of channels in a gamma emission spectrum which is grouped to identify an isotope or for some other purpose, eg for backgrounds. A peak ROI, such as that for ^{210}Pb will include several channels either side of the peak centre, typically 4 channels in 8192 channel spectrums. Background ROIs for some isotopes may not be located directly adjacent to the peaks ROIs due to interference from other isotope peaks. In this case the background ROI is then moved further away from the peak ROI to an area which still reflects the typical background on that side of the peak.

Relative efficiency - is a measure of a germanium detector’s efficiency compared to 3’ x 3’ NaI detector. Relative efficiencies range from 10% to over 150%.

Resolution – is a numerical index of at least one quality of performance, the ability to detect and present results with the least distortion. While all of an electronic system might affect resolution, for gamma counting the reality is that the detector assembly is the principal element in determining resolution. In a modern gamma counter, if the detector assembly exhibits good performance, ie low resolution, then the entire instrument will likely give good performance. Resolution is normally expressed in terms of '%FWHM' or 'Percent of Full Width at Half Maximum'. See also 'FWHM.'

Standards and source materials – are of similar matrix to the samples to be analysed with accurately known amounts of specific radionuclides. Standards are always prepared in the same geometry, ie shape and container, and matrix as the samples to be counted. Standards are not highly radioactive, but their activity should be accurately known and traceable to a certified source material. Suppliers of source materials provide a value for the activity, the uncertainty of the activity value and equilibrium status of the progeny. See also 'Calibration a detector' and 'Backgrounds – matrix backgrounds'.

Systematic error (or systematic uncertainty) – are predictable variations from the true value caused by a particular instrument or experimental technique. See also 'Random error', 'Precision' and 'Accuracy'.

Temperature cycling – is the warming up of a detector to allow a fresh cool down cycle. The warm up period must always be long enough to allow a complete warming of the whole detector assembly to room temperature. This may take several days. Cooling should only be done with enough liquid nitrogen to completely fill the dewar as the nitrogen boil off rate will be high during the initial cooling period. Traps known as molecular sieves scavenge remaining air and moisture from within the vacuum space during the cooling cycle.

Total propagated uncertainty (TPU) – the summed uncertainties associated with an activity result, usually reported in the same units as the activity, however, TPU may also be expressed as a percentage. The calculations for uncertainties are taken from the document 'Quantifying uncertainty in analytical measurement', Eurachem/CITAC Guide 2nd edition, and are usually expressed in the same units as the activity, to ± 1SD.

Uncertainty – a parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to that measurement. Uncertainty of measurement does not imply doubt about the validity of a measurement, on the contrary, knowledge of the uncertainty implies increased confidence in the validity of a measured result.

13 References

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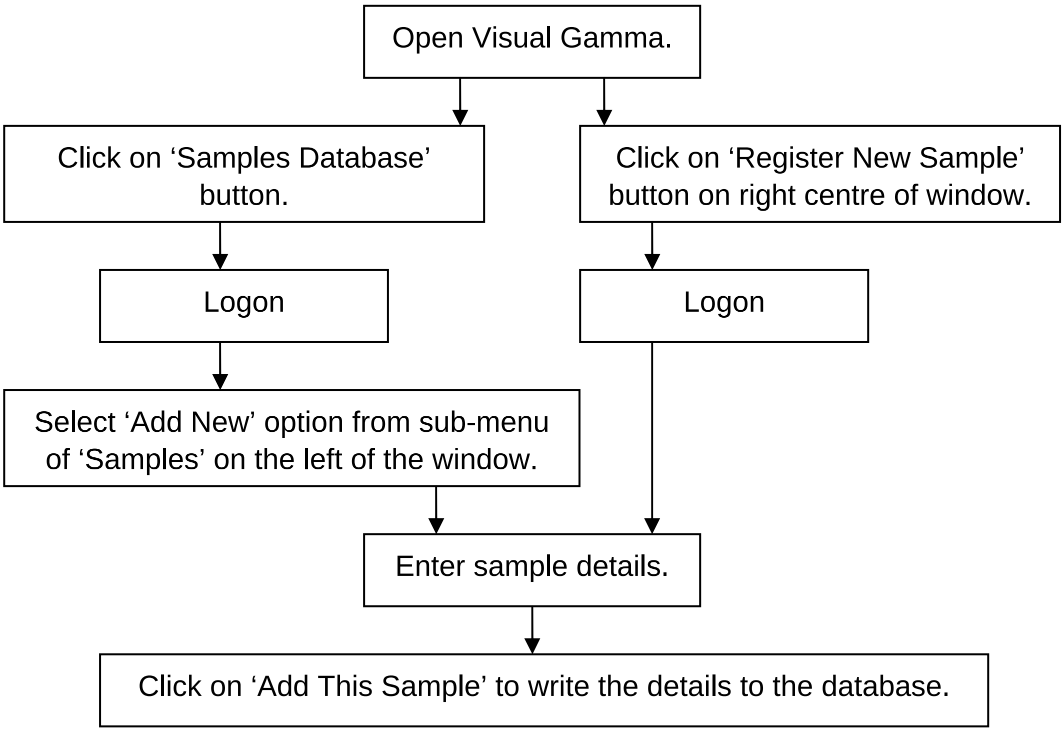
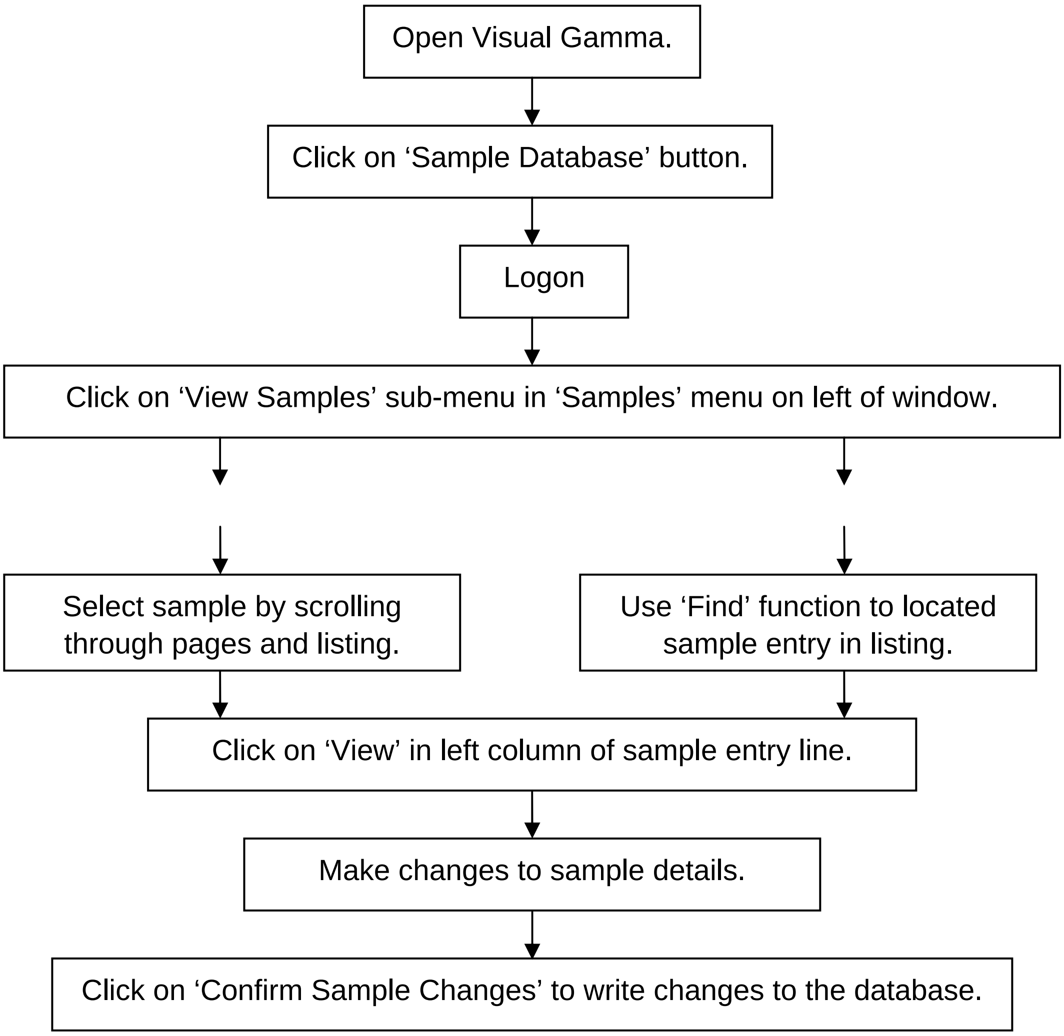
Appendix A Program structure flow charts

See ‘Visual gamma analysis operations manual’ (Esparon & Pfitzner, in prep) for complete information on operational procedures.

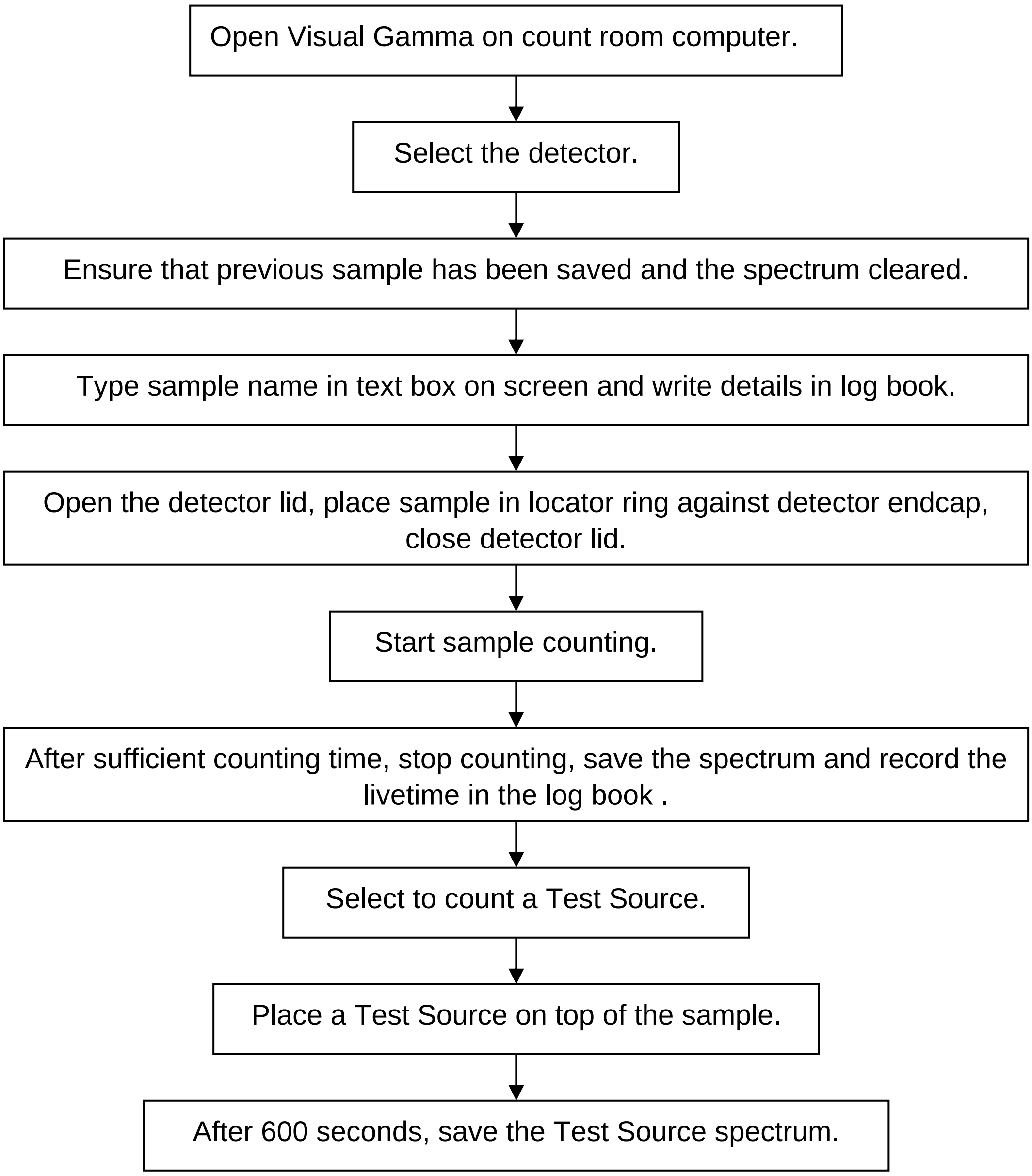
A1 Entering sample details in database



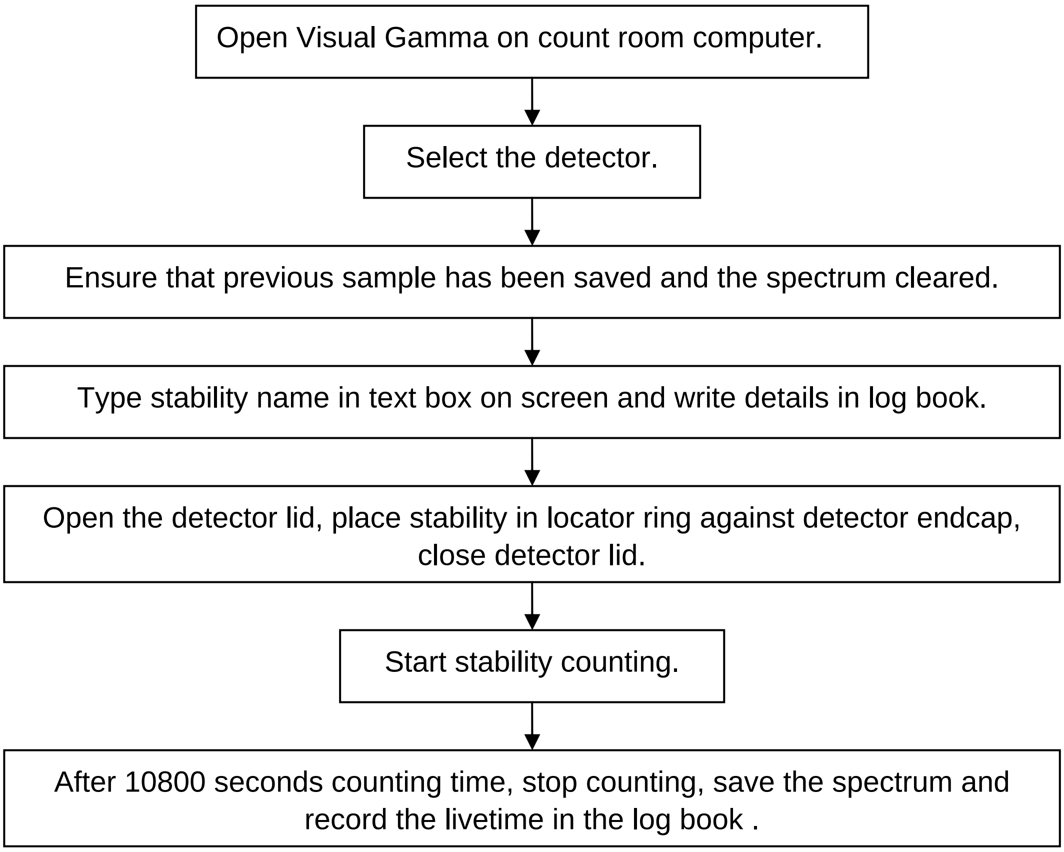
A.2 Changing sample details in database



A.3 Counting a sample, standard or background and saving the spectrum



A.4 Counting a stability check source



Open Visual Gamma on count room computer.

Select the detector.

Ensure that previous sample has been saved and the spectrum cleared.

Type stability name in text box on screen and write details in log book.

Open the detector lid, place stability in locator ring against detector endcap, close detector lid.

Start stability counting.

After 10800 seconds counting time, stop counting, save the spectrum and record the livetime in the log book .

Appendix B Peak shift calculations

The adjustment of the ROI channel numbers uses the following formulae:

A slope of the change in channel number is calculated from the Test Source 186 and 1001 keV locations (which should reflect those in the sample spectrum), compared to the calibrated values:

$$Slope = \frac{((T1001 - C1001) - (T186 - C186))}{(T1001 - T186)}$$

where

C = calibrated channel number for peak centre,

T = test source channel number for peak centre.

The offset value is determined,

$$Offset = (T186 - C186) - Slope * T186$$

And the amount to shift the individual ROI channels is,

Shift = (Slope*channel number from cal file) + Offset

Therefore the new channel number is,

New ROI channel number = INTEGER(channel number from cal file – Shift)

This energy calibration is required for all sample analysis, background analysis or standard analysis/calibration procedures.

Appendix C Sources of uncertainty and propagation of uncertainty equations

Table C1 Uncertainty calculations for mathematical manipulation in *Visual Gamma*

Function[a]	Manipulation	Uncertainty[b]
$y = a_1 x_1 - a_2 x_2$	Addition/subtraction	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = a_1 x_1 + a_2 x_2$	Addition/subtraction	$s_y = \sqrt{a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2}$
$y = a_1 x_1 x_2$	Multiplication/Division	$\frac{s_y}{y} = \sqrt{\left(\frac{s_{x_1}}{x_1}\right)^2 + \left(\frac{s_{x_2}}{x_2}\right)^2}$
$y = a_1 \left(\frac{x_1}{x_2}\right)$	Multiplication/Division	$\frac{s_y}{y} = \sqrt{\left(\frac{s_{x_1}}{x_1}\right)^2 + \left(\frac{s_{x_2}}{x_2}\right)^2}$
$y = x^m$	Power of *x*	$\frac{s_y}{y} = \frac{m s_x}{x}$
$y = e^{-ax}$	Power of *e*	$\frac{s_y}{y} = a s_x$
$y = f(x_1, x_2)$	Function of two variables	$s_y = \sqrt{\left(\frac{\partial f}{\partial x_1}\right)^2 s_{x_1}^2 + \left(\frac{\partial f}{\partial x_2}\right)^2 s_{x_2}^2}$

[a] a, a_1, a_2 and m represent constants, x, x_1 and x_2 are independent variables and y is the dependent variable.

[b] s_i is the estimated standard deviation in variable x_i.

The principal components of the combined uncertainty in a measured gamma-ray emission rate are those associated with

a) calibration standards
b) measurement of the peak areas
c) reproducibility of the sample detector geometry
d) pulse pile-up
e) coincidence summing correction
f) decay corrections during and prior to counting
g) absorption correction when sample matrix differs from that of the standard
h) detector full energy peak efficiency interpolation
i) subtraction of instrument and matrix background counts from sample or standard.

Appendix D Equations for the calculation of weighted average

Consider the following values and their associated uncertainties are $b \pm \delta b$, $c \pm \delta c$ and $d \pm \delta d$, then weighted mean W_m is

For example 0.23 ± 0.06, 0.30 ± 0.09, 0.28 ± 0.055

$$W_m = \frac{\frac{b}{\delta b^2} + \frac{c}{\delta c^2} + \frac{d}{\delta d^2}}{\frac{1}{\delta b^2} + \frac{1}{\delta c^2} + \frac{1}{\delta d^2}}$$

$W_m = 0.264$

and the propagated uncertainty in the weighted mean is

$$\Delta W_m = \frac{1}{\sqrt{\frac{1}{\delta b^2} + \frac{1}{\delta c^2} + \frac{1}{\delta d^2}}}$$

$\Delta W_m = 0.037$

The above two equations for weighting are expressed as the weighted average

$$\mu_x = \frac{\sum_{i=1}^{n} w_i x_i}{\sum_{i=1}^{n} w_i}$$

$\mu_x = 0.264$

And Internal Variance:

$$s^2(\mu_x) = \frac{1}{\sum_{i=1}^{n} w_i}$$

$s^2(\mu_x) = 0.00137$ ($s(\mu_x) = 0.037$)

where $w_i = 1/s_i^2$ and s_x^2 is the sample variance (the standard deviation of the sample is the square root of the variance), in ANSI N42.14-1999 ‘American National Standard Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides’.

Appendix E Peak area and uncertainties by end point averaging

From Canberra Product Catalogue Edition Twelve, page 25, consider the below sample spectrum. As the background is similar to the peak and variable, the determination of continuum background is best made by averaging over several channels. A is the net area of the peak, P is the sum of the counts in the peak ROI of n channels and B_1 & B_2 are the backgrounds summed over n_1 & n_2 channels respectively.

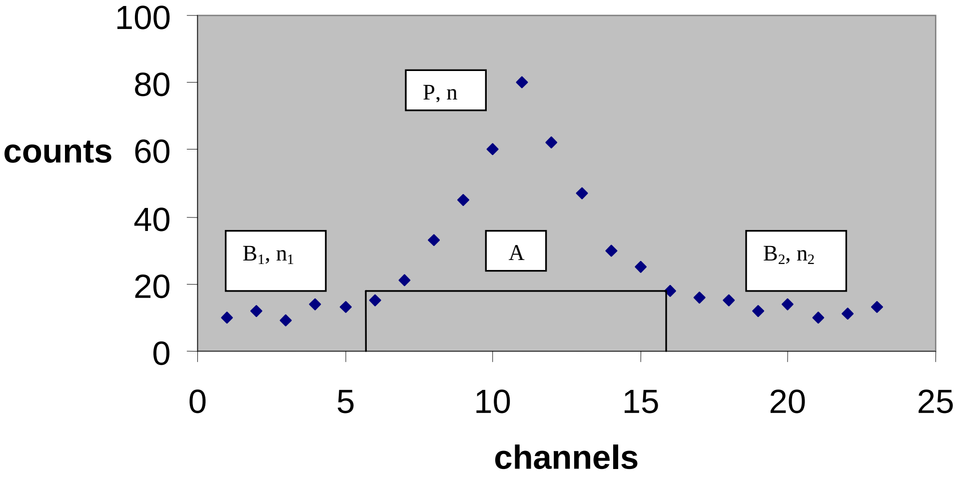


Figure E1 Typical peak with continous background

For example, in the above typical ***sample*** peak, the counts in channels 1 to 23 are 10, 12, 9, 14, 13, 15, 21, 33, 45, 60, 80, 62, 47, 30, 25, 18, 16, 15, 12, 14, 10, 11, 13. Peak channels are 6 to 16 (11 channels), LBG 1 to 5 (5 channels) and RBG 17 to 23 inclusive (7 channels).

Therefore, the sample peak area is

$$A = P - \frac{n}{2}\left(\frac{B_1}{n_1} + \frac{B_2}{n_2}\right)$$

A = 436 – 11/2(58/5 + 91/7) = 300.7

And the standard deviation

$$\sigma A = \sqrt{\left[P + \left(\frac{n}{2}\right)^2 \left(\frac{B_1}{n_1^2} + \frac{B_2}{n_2^2}\right)\right]}$$

σA = √(436+(11/2)^2 (58/25+91/49)) = 23.7

The same end point averaging equations are used to calculate the net area and net area uncertainties in the background spectra, the radionuclide standards and in the samples.

If the typical corresponding instrument ***background*** for those channels was 2, 4, 4, 5, 4, 6, 5, 7, 6, 9, 6, 8, 5, 3, 4, 5, 4, 2, 4, 5, 3, 4, 4, then the instrument background count net peak area and associated uncertainty is 22.7 ± 10.2.

Net Peak Area (only Compton background corrected):

Assume that the sample above was counted for 80 ksec and the background for 160 ksec. Dividing each by their respective count times, the typical sample peak is 3.76 ± 0.28 counts/ksec and instrument/matrix background is 0.142 ± 0.064 counts/ksec.

Uncertainty in net peak area when subtracting the instrument/matrix background:

The above ANSI N42.14-1999 section on uncertainties provides the following equation for subtracting the instrument/matrix background from Compton corrected peak area:

$$s_y = \sqrt{(a_1^2 s_{x_1}^2 + a_2^2 s_{x_2}^2)}$$

Subtracting the instrument background from the typical peak gives a result of 3.62 ± 0.29 counts/ksec, using this equation.

The estimate of uncertainty in net peak area when subtracting the instrument/matrix background in this case is 0.29, provided by $s_y = \sqrt{s_{x1}^2 + s_{y2}^2}$.

This is the calculation used in Visual Gamma.

Appendix F Minimum detectable activity (or lowest limit of detection)

Currie’s 1968 derivation of MDA is (From Canberra Product Catalogue Edition Twelve, page 27):

$$MDA_{(Bq/unitwt)} = \frac{2.71 + 4.66(\sigma)}{T * (Eff) * Y * (wt)}$$

where

σ: standard deviation of the background counts at the energy of interest,

T: collection time (ksec),

Eff: efficiency at the energy and geometry of interest,

Y: branching ratio and

Wt: sample weight.

The branching ratio *Y* is taken into account in the ***eriss*** calibration process and included in the calculation of the efficiency, hence the term *Y* is redundant.

In the following consider the isotope peak for ^{210}Pb at 46.5 keV on detector O. A blank instrument background count for on average of 280 ksecs results in a standard deviation of the background of σ_B = 0.2 counts/ksec. This amounts to 17 counts in 86 ksec. Consequently, the MDA, using 17 counts in the background peak over 86 ksecs, is 11 Bq/kg.

Practical experience would indicate that 12 Bq/kg are realistic expectations of ^{210}Pb MDA (95%) in 15g sediment samples with blank instrument backgrounds respectively counted for one day. If instrument or matrix backgrounds are well known, their uncertainties are significantly reduced and the length of the sample count time and the Compton background of the sample will become the major contributors to the MDA.

Appendix G FWHM (full width half maximum) calculation

The FWHM of a specified peak is a measure of the resolution of the detector, a value of the ability of the detector to resolve, or differentiate between, two nearby peaks. The smaller the FWHM, the better the detector resolution. FWHM is expressed in terms of energy, in the case of the HP germanium detectors, as keV. An approximate rule of thumb is that one should be able to resolve two energies that are separated by more than one value of the detector FWHM at that place in the energy spectrum. The FWHM increases with increasing energy.

Method of calculating FWHM: after subtraction of the continuum baseline from the gross counts in the peak, determine the number of channels (to within 0.1 channels) above the half-height of the peak and convert that number of channels to energy. For simplicity, assume linear interpolation between each margin channel which may be a fractional portion of the FWHM.

For example, in the above typical peak, the counts in channels 1 to 23 are 10, 12, 9, 14, 13, 15, 21, 33, 45, 60, 80, 62, 47, 30, 25, 18, 16, 15, 12, 14, 10, 11, 13.

Peak channels are 6 to 16, LBG 1 to 5 and RBG 17 to 22 inclusive. The average Compton background is 12.5 counts/channel. Therefore net maximum counts are 80-12.5 = 67.5, and half maximum is 33.75 counts.

Net counts within peak are 2.5(6), 8.5(7), 20.5(8), 32.5(9), 47.5(10), 67.5(11), 49.5(12), 35.5(13), 17.5(14), 12.5(15) and, 5.5(16).

The half maximum value falls between the channels 9, 32.5 and 10, 47.5 on the left side and between channels 13, 35.5 and 14, 17.5 on the right side.

Treating the left side, 47.5-32.5 = 15, 33.75-32.5 = 1.25, 1.25/15 = 0.08, therefore left channel is 9.08, rounded to 9.1.

On the right side of the peak, 35.5-17.5 = 18, 35.5-33.75 = 1.75, 1.75/18 = 0.09, therefore right channel is 13.09, rounded to 13.1.

Thus FWHM channels are 9.1 to 13.1, 5 channels. If the keV/channel is 0.19, then FWHM = 5*0.19 = 0.95 keV.

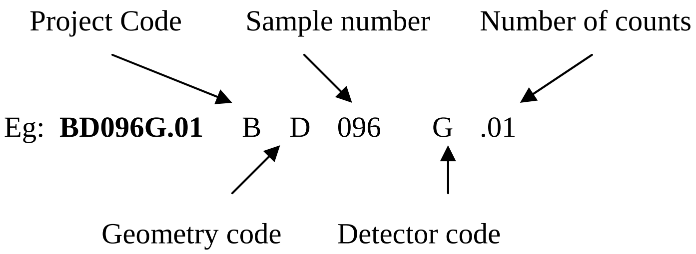
The FWHM is routinely calculated on several peaks spread over the energy spectrum (46.5, 186 and 911 keV), using data from stability standard counting. Stored data for the FWHM is displayed for each detector to allow visual checking of any change in resolution over time, a part of the QA measures.

Appendix H Sample and analysis coding

All samples, backgrounds and standards are designated a Project code.

Standards are identified as ‘Z’ and *backgrounds* as ‘Y’ in the initial (Project) character of their spectrum filename. The initial character of a *sample* spectrum filename may have any remaining character in the alphabet to designate a project code. Current projects and associated alphabetic codes are listed in Table H1.

All samples, backgrounds and standards are prepared by placing the material to be analysed in specially designed plastic containers or by casting in polyester resin. The containers and polyester resin moulds are designated ‘geometries’ and are given a second alphabetic code – see Table H2. The second character of the sample/ standard/background spectrum filename identifies the geometry of the sample. This geometry code provides a key for the program to choose the correct efficiency calibration data, as the detector efficiency varies with sample geometry.



Project codes

Table H1 Project codes

Code	Project Code
A	Building materials
B	Mussels
C	Artificial ponds
D	MnO_2 - Acrylic Fibre Filters
E	Aboriginal food items
F	Vegetable material
G	Adsorption/Desorption experiments
I	Surface waters
J	Sediments/soils
K	MnO_2 and FeOH Precipitations
N	Dating
O	Underground waters
Q	Land Application
X	Miscellaneous single samples
Y	Background standards
Z	Active standards and other calibration samples

Geometry codes

Table H2 Geometry codes

Code	Geometry Code
A	Organic disc
B	Inorganic disc
C	Organic cup
D	Inorganic cup
E	Organic Marinelli
F	Inorganic Marinelli
Q	Medium pressed sediment
R	Large pressed soil
V	Inorganic small disc
X	Non-standard geometry
Y	Counting with no sample

Sample Number

Incremental press code to uniquely identify a sample to be analysed.

Detector Codes

Refer Appendix I for detector specifications

O – Detector O

N– Detector N

X– Detector X

Number of counts

Starting at .01 it represents the number of times a sample has been counted on a particular detector.

Appendix I Detector specification and performance sheet

I.1 Detector O

Delivery:	1/5/1998

General information

Manufacturer:	Ortec
Type:	
Model:	GMX-20190-S
Serial Number:	37-N11294A

Cryogenic information

Configuration:	LLB-GMX-HJ
Dewar Model:	30S
Dewar capacity:	30 litres
Static holding time:	10 Days
Cool down time:	24 hours

Crystal dimensions

Diameter:	52.3 mm
Length:	45.9 mm
End cap to crystal:	3 mm

Electrical characteristics

Bias voltage:	-3500Volts
Preamp Model:	120-6B
Preamp serial number:	108

Resolution and efficiency

Date: 31-3-1998	**Warranted**	**Measured**	**Time Constant**
Resolution (FWHM) at 1.33 MeV, ^{60}Co:	1.90 keV	1.78 keV	6 µs
Peak to Compton Ratio, ^{60}Co:	48	48	6 µs
Relative efficiency at 1.33 MeV, ^{60}Co:	20.0%	21.0%	6 µs
Peak Shape (FWTM/FWHM), ^{60}Co:	1.95	1.87	6 µs
Peak Shape (FWFM/FWHM), ^{60}Co:	2.80	2.52	6 µs
Resolution (FWHM) at 5.9 keV, ^{55}Fe:	690eV	489eV	

I.2 Detector N

Delivery:	9/5/2000

General Information

Manufacturer:	Ortec
Type:	
Model:	GMX-20190-S
Serial Number:	40-N31423B

Cryogenic information

Configuration:	LB-GMX-HJ-S
Dewar Model:	30B
Dewar capacity:	30 litres
Static holding time:	10 Days
Cool down time:	24 hours

Crystal dimensions

Diameter:	56.2 mm
Length:	51.9 mm
End cap to crystal:	3 mm

Electrical characteristics

Bias voltage:	-4000 Volts
Preamp Model:	120-6
Preamp serial number:	160

Resolution and efficiency

Date: 9-5-2000	**Warranted**	**Measured**	**Time constant**
Resolution (FWHM) at 1.33 MeV, ^{60}Co:	1.90keV	1.77keV	6 µs
Peak to Compton Ratio, ^{60}Co:	48	52.5	6 µs
Relative efficiency at 1.33 MeV, ^{60}Co:	20.0%	26.2%	6 µs
Peak Shape (FWTM/FWHM), ^{60}Co:	1.95	1.82	6 µs
Peak Shape (FWFM/FWHM), ^{60}Co:	2.80	2.38	6 µs
Resolution (FWHM) at 5.9 keV, ^{55}Fe:	690eV	488eV	

I.3 Detector X

Delivery: 9th September 2010

General Information

Manufacturer:	Ortec
Type:	Pop-Top with X-Cooler II
Model:	GEM40P4-75
Serial Number:	50-TP12839A

Cryogenic Information

Configuration:	Pop-Top
Dewar Model:	no dewar, X-Cooler II
Dewar capacity:	n/a
Static holding time:	n/a
Cool down time:	24 hours

Crystal dimensions

Diameter:	63.6 mm
Length:	59.7 mm
End cap to crystal:	4 mm

Electrical characteristics

Bias voltage:	+ 4000 V
Preamp Model:	A257P
Preamp serial number:	10154405

Resolution and efficiency

Date: 9-5-2000	**Warranted**	**Measured**	**Time constant**
Resolution (FWHM) at 1.33 MeV, ^{60}Co:			
Peak to Compton Ratio, ^{60}Co:	64.1	75.1	6 µs
Relative efficiency at 1.33 MeV, ^{60}Co:	40%	45%	6 µs
Peak Shape (FWTM/FWHM), ^{60}Co:	1.9	1.9	6 µs
Peak Shape (FWFM/FWHM), ^{60}Co:	2.6	2.5	6 µs
Resolution (FWHM) at 5.9 keV, ^{55}Fe:	870eV	689eV	6 µs

Appendix J Matrix attenuation correction equations

NOTE: Attenuation correction has not yet been implemented.

Two methods of measuring the matrix attenuation correction are employed:

1 counting of a test source or similar high activity gamma emitter placed both above a standard and then above the sample, and

2 counting a uniformly spiked unknown sample matrix with an accurately measured aliquant of gamma emitting radionuclides of interest in the same geometry as standards used to calibrate the detector.

Method 2 provides the actual internal attenuation, which must be applied to Method 1), thereby allowing the routine use of Method 1).

Method 1 is used routinely at the time of counting, employing the 600 second test source count to give the attenuation correction for most geometries except R, which requires a 2000 second count time with a Test Source.

In this case, the attenuation correction C_a at energy E is given by

$$C_a(E) = \frac{[n_s(E)] - B_s(E)]}{[n_u(E) - B_u(E)]} \cdot k$$

where

$n_s(E)$ is the net count rate of the Test Source in the peak areas of the standard (Flying Fox sand) at energy E,

$n_u(E)$ is the net count rate of the Test Source in the peak areas of the sample at energy E,

$B_s(E)$ is the net count rate in the peak areas of the background spectra associated with the standard (Flying Fox sand),

$B_u(E)$ is the net count rate in the peak areas of the background spectra associated with the sample.

(k) is the factor to convert the Test Source transmission ratio to FF sand to internal attenuation factor.

Notes

1. These are all net counting rates with only continuum background subtracted.
2. The sample background spectra being the sample counts (adjusted to 600 sec).
3. The standard background spectra being the count of FF sand or equivalent matrix (adjusted to 600 sec).
4. The above assumes a standard weight of sample is used – the formula does not hold for different weights.

Test Sources activities spectra were checked at 46.5, 63, 186 and 1001 keV. Due to the low counts rates for the 1001 keV line in 600 seconds, the count to count variation on the same sample was large (> 5%). Therefore the 1001 keV energy was unsuitable for use in the attenuation calculation process. The 46.5, 63 and 186 keV lines were of sufficiently high count rate to have small statistical uncertainties (generally < 1%) and are used in the attenuation correction calculations.

The 186 keV line showed only 5% attenuation in a 52% iron oxide sediment which had 50% greater attenuation than the Flying Fox sand for the 46.5 keV line. Thus the 186 keV line would be suitable, in nearly all cases, as a measure of the unique un-attenuated count rate of the Test Source.

Attenuation is highly energy dependent. The following isotope lines are affected:

^{212}Pb @ 40 keV,

^{210}Pb @ 46.5 keV,

^{214}Am @ 59 keV

^{234}Th @ 63 keV,

^{230}Th @ 67 keV and

^{226}Ra @ 144 keV.

As attenuation correction coefficients are only calculated for the 46.5 and 63 keV energies, coefficients at the other energies must be extrapolation & interpolated, using the 186 keV attenuation if necessary.

Example for matrix attenuation correction calculations

All Test Sources have been counted to establish their activities. The results for the net counts (Maestro, using ZQ000SS.roi) on detector S, collimated through an empty Q geometry were:

Table J1 Activities of current test sources on detector S

Energy	TS ‘F’	TS ‘G’	TS ‘K’	TS ‘L’
46.5	13941 ± 155	1461 ± 57	3291 ± 79	3583 ± 82
63	36796 ± 228	9963 ± 129	10719 ± 130	11246 ± 132
186	27252 ± 180	9182 ± 113	9311 ± 112	10007 ± 115

New Test Sources will be prepared with ^{210}Pb and ^{238}U to activities comparable to Test Source ‘F’.

As Flying Fox sand is the most commonly used matrix for the preparation of standards, the attenuation coefficients were calculated from the ratio of the sample Test Source (TS) transmission against the TS transmission through Flying Fox sand.

The following sample types were used to determine the attenuation for detector S, geometry Q:

Empty Q container (ZQ000)

Flying Fox sand (ZQ059)

Barite, external sample XX05006 (JQ648) [18g]

Feldspar, external sample XX05007 (JQ649)

42% Quartz, external sample XX05008 (JQ650)

52% Iron oxide, external sample XX05015 (JQ657)

12.5% Fe powder in FF sand (ZQ086)

25% Fe powder in FF sand (ZQ087)

Flying Fox sand + BL-5 (ZQ075)

Flying Fox sand + BL-5 (ZQ076)

Barite, external sample XX05006 + BL-5 (ZQ071)

Feldspar, external sample XX05007 + BL-5 (ZQ072) [12g]

42% Quartz, external sample XX05008 + BL-5 (ZQ073)

52% **Iron oxide,** ***external sample XX05015 + BL-5 (ZQ074)***

12.5% Fe powder in FF sand + BL-5 (ZQ088)

25% Fe powder in FF sand + BL-5 (ZQ089)

Unless otherwise noted, all samples were loaded to the standard weight for geometry Q of approximately 15g.

For future detector attenuation calibrations, count the above standards & samples shown in ***bold italics*** for at least 60 ksecs. Count all of the ***bold italics*** and those only in **bold** with the Test Source. Note that many of these are standards which will be counted during the calibration process and do not need to be counted again for the attenuation determination. For ‘R’ geometry, a 6% Fe powder matrix should provide an additional attenuation point at a relevant attenuation for that geometry.

Several materials (sodium sulphate, sugar and calcium phosphate) were tested in an attempt to find a material with less attenuation than Flying Fox sand. A 10g sample of ground white sugar showed 17% higher transmission than FF sand @ 46.5 keV and 8% more @ 63 keV, with individual counting uncertainties of 1.7% and 1.1 respectively.

If the standard or sample has sufficient activity in these energies (> 1% of transmission counts), then these must be measured and subtracted from the TS transmission data as counts/ ksec.

The net peak area (- Compton background) in counts/ ksec in each energy, 46.5, 63 & 186 keV, are compared to the similar peak areas for Flying Fox sand, both for Test Source transmission and for internal attenuation. Spectral data and plots showing the relationships between the Test Source attenuation and the internal attenuation for each energy are in Excel file ‘Collimated Attenuation.xls.’ When establishing the internal attenuation, the activity of the standard must be known accurately so that the relationship between the various matrices can be calculated as counts/ ksec/ Bq.

The attenuation coefficient for both the 46.5 and 63 keV is dependent upon the amount of attenuation.

For example, for detector S, geometry Q at 46.5 keV

Table J2 Example values for attenuation correction

Transmission TS through sample : Transmission TS through FF sand	Actual internal attenuation (BL-5)	(*k*) from above formula
0.502	0.793	1.58
0.628	0.774	1.23
0.819	0.880	1.07
0.838	0.900	1.08

Steps in the attenuation correction process are:

1. count Test Source above sample
2. determine net counts/ ksec of Test Source transmission in the 46.5, 63 & 186 kev peak areas
3. divide the sample net counts/ ksec TS transmission by the equivalent FF sand TS transmission in counts/ ksec
4. use the factor *(k)* to find the actual internal attenuation (AIA)
5. apply the AIA correction to the sample to give the attenuation corrected activity.